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Physicochemical, Morphological and Anticorrosive Properties of Electrodeposited ZnNi Alloy Coating

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Abstract: Corrosion in its many forms is a major problem for many types of materials, extending from machining to manufacturing and daily use. In fact, it affects practically all engineering projects, from the biggest to the smallest: energy production, construction, transport, medical sector, electronics, etc. Consequently, corrosion generates both economic and environmental problems. To prevent corrosion and preserve materials' durability, several protection methods can be applied. These included protection by applying a coating on the metal surface. In this context, Zinc based alloy coatings have attracted much interesting scientific community because of their excellent mechanical and anti-corrosive properties. In the present work, ZnNi coatings were electrodeposited on steel substrates using a chloride bath containing ammonium salts. The effect of some experimental parameters, namely, potential and metal ion concentration ratio on the deposition kinetics of ZnNi system as well as on its physicochemical, morphological and anticorrosive properties has been investigated. For this purpose, an appropriate experimental work was performed by using both electrochemical (cyclic voltammetry, chronoamperometry, open circuit potential, potentiodynamic polarization) and non-electrochemical (SEM-EDX) analysis methods. Based on the obtained results, it has been revealed that simultaneous codeposition of both elements Zn and Ni is possible in our experimental conditions. However, deposition kinetics exhibited a strong dependence on explored parameters. Also, morphological aspect and chemical composition of deposits are strongly influenced by metal ion concentration ratio. Investigation of corrosion behaviour confirms the protective effect of coatings acted as a sacrificial anode. Furthermore, increasing Ni content in the deposits induces a significant enhancement in corrosion resistance. Thus, Zn-Ni alloy coatings prepared from the Ni(II)-rich bath exhibited better corrosion resistance.

Keywords: Corrosion, Steel, ZnNi alloy coating, Electrodeposition

Introduction

Nowadays, corrosion phenomena have taken a considerable importance due to the increasing use of metals and alloys in modern life. It is considered to be a major source of metal structures and industrial equipments failure. From an economic viewpoint, the corrosion consequences are quite heavy and may even be disastrous (Chevalier,2010). These include: work and manufacturing interruptions, higher maintenance costs, risk of injury or accident to personnel, environmental contamination (Rodriguez, 2021). To prevent corrosion and reduce damage, various protection methods have been developed, including application of coatings, cathodic protection, and inhibition protection, etc (Venkatesha, 2008).

Steel substrates are generally protected against corrosion by a sacrificial electrodeposited coating. Zinc-based alloys and cadmium coatings (Lin & Sriraman 2006), are some of the most commonly sacrificial coatings. Cadmium coatings are considered highly reliable due to their excellent corrosion resistance and engineering properties (Bashkov, 2005). However, they are highly toxic. So, their manufacturing processes require cyanide baths, which are also characterised by their high toxicity. Furthermore, cadmium plating induces a significant amount of hydrogen into the substrate which causes its weakening. In recent years, increasing environmental and ecological preoccupations requires the development of an alternative coating that can efficiently substitute cadmium. In this context, zinc deposits show excellent sacrificial behaviour (Pistofidis,2006). However, their application remains limited in the automotive industry because of their high dissolution rate and poor mechanical properties (Tomic, 2023)

To this end, the development of zinc-based alloys is a highly attractive strategy for enhancing pure zinc coatings' protective properties (Silva, 2010). This is therefore based on addition of some elements to zinc matrix, which can form a very wide range of metallic combinations of ZnX type, such as ZnCo (Bhat, 2022), ZnCu (Oulmas, 2019), ZnFe (Ebrahimi, 2003), ZnMn (Bedir, & Fashu, 2015). Among these coatings, ZnNi alloy appears to be a potential candidate due to its viability as an alternative to cadmium coatings. To this end, Zn-Ni alloys containing 15 to 20% weight of nickel exhibited a very high corrosion resistance compared to cadmium and titanium coatings.

Such coating is widely applied in the aerospace and energy production fields due to its excellent resistance at high temperatures and even under severe oxidation conditions (Abou-Krisha, 2007). In recent years, an extended research activity is focused on the electrodeposition and characterisation of ZnNi alloy coatings (Pedroza,2014), The goal is to optimise deposition parameters in order to develop new coatings which provide increasingly enhanced anticorrosion properties (Roventi, 2015).

This work is situated in this context of ideas. In particular, this study focuses on the electrodeposition of ZnNi alloy coatings from a chloride bath containing ammonium salts. The effect of some experimental parameters, such as potential and Ni^{2+} ion concentration on deposition kinetics of ZnNi alloys coatings, physicochemical properties, surface morphology and corrosion behavior were investigated.

Method

Electroplating Kinetic

The electrochemical experiments were carried out in a conventional three-electrode cell. A vitreous carbon rotating disk electrode (RDE) (0.2 cm^2) with a rotation speed fixed at 250 rpm was used as working electrode to investigate the kinetic of ZnMn electrodeposition. The counter electrode was a platinum wire immersed in a separate compartment containing solution without electroactive metallic cations. All potential values were measured with respect to a saturated calomel reference electrode $Hg/Hg_2Cl_2/Cl^-$ (SCE). All experiments were performed at ambient temperature in an aerated chloride bath containing ammonium chloride. The latter improves bath conductivity and also complexes metal cations (Zn^{2+} and Ni^{2+}), this avoids the formation of hydroxides during ZnNi electroplating. To carry out this work, three baths were used, which are differentiated by the metal ion concentration ratio, their compositions are shown in Table 1. The pH was adjusted to 2 by adding dilute hydrochloric acid solution. The solutions were prepared immediately prior to each experiment.

Table 1. The composition of electroplating baths

Bath	Ratio $[Ni^{2+}]/[Zn^{2+}]$	Species	Concentration (mol/l)
1	0.66	$NiCl_2$, H_2O	0.2
		$ZnCl_2$, 6 H_2O	0.3
		NH_4Cl	2.8
2	1	$NiCl_2$, H_2O	0.3
		$ZnCl_2$, 6 H_2O	0.3
		NH_4Cl	2.8
3	1.5	$NiCl_2$, H_2O	0.45
		$ZnCl_2$, 6 H_2O	0.3
		NH_4Cl	2.8

Preparation of Steel Substrates and Elaboration of ZnNi Coatings

Steel substrates with area of 1 cm^2 were used to elaborate Zn-Ni alloy coating. Prior to each experiment, steel substrates were polished mechanically with a METKON FORCIMAT polishing machine equipped with a rotating disk using abrasive paper with various granulometry (400, 600, 800, 1000, 2000, 4000) and then lustrated on a diamond-paste impregnated felt with different grains size (9 µm, 3 µm and 1 µm). Then, they were degreased with ethanol for 10 minutes using ultrasound. Finally, rinsed with distilled water and dried.

Corrosion Test Solutions

Corrosion behaviour of ZnNi alloy coatings was studied in 3% weight of NaCl solution

Characterizations Techniques

The electrochemical measurements were performed using an EG&G 273A Potentiostat/Galvanostat controlled by a microcomputer via GPIB interface operated by M352 EG&G software. Cyclic voltammetry was used to study deposition kinetics and chronoamperometry was employed to prepare ZnNi samples. The anti-corrosion properties of ZnNi coatings were evaluated by using various electrochemical methods, such as open circuit potential (OCP), linear polarization and Tafel curves.

The morphological properties of ZnNi alloy coatings were studied by scanning electron microscope (ESEM XL 30 Philips).The chemical compositions were measured by SEM-JEOL JSM-7610 scanning electron microscope coupled with X-ray microanalysis (EDX).

Results and Discussion

Electrochemical Study

Electrodeposition of Zn and Ni

Before investigating the electrochemical deposition kinetics of ZnNi, a voltammetric study of deposition kinetics for each element was performed separately. Figure 1.a shows the cyclic voltammograms recorded on a vtreous carbon electrode RDE from a zinc chloride bath ($[ZnCl_2]$ = 0,3 mol/l , $[NH_4Cl]$ = 2,8 mol/l , pH = 2). The results show a cathodic current starting at -1.08 V vs. SCE, it is attributed to the reduction of Zn^{2+} ions.(zinc metal deposit). By scanning toward more cathodic potential, an increase in current density is observed, suggesting the proton reduction (evolution of hydrogen) (Fashu, 2015).

So, the reduction current density increases with the potential, this indicates an increase of the deposit rate. In the reverse scan, one dissolution peak is observed at -1 V vs. SCE. It corresponds to zinc dissolution. This peak becomes more extensive and intense as the applied cathodic potential increases. This can be explained by a considerable increase in the amount of zinc deposited.

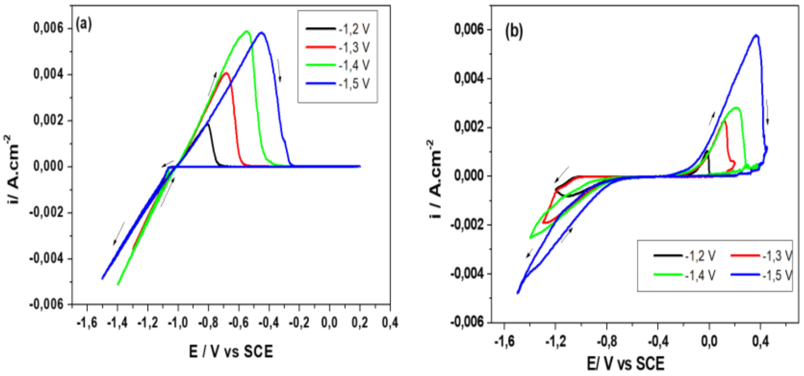

Figure 1. Cyclic voltammograms obtained on vitreous carbon RDE at various cathodic limit potentials, pH = 2, V_b = 20 mV/s, w = 250 rpm, (a) zinc voltammograms, (b) Ni voltammograms

The electrochemical study of nickel deposition kinetics from a nickel chloride bath ($[NiCl_2]$ = 0,3 mol/l , $[NH_4Cl]$ = 2,8 mol/l , pH = 2) was also performed on a vitreous carbon electrode RDE. Figure 1.b illustrates the cyclic voltammograms obtained at several cathodic limit potential. All recorded cyclic voltamperomograms show a cathodic current at -0.85 vs. SCE which corresponds to the Ni^{2+} reduction (formation of the nickel deposit). At more cathodic potentials, a strong increase in current density is noted, which can be explained by the appearance of hydrogen evolution, that occurs simultaneously with nickel deposition. In the anodic region of voltammograms, a quite symmetrical peak is observed around -0.3 vs. SCE, which is attributed to nickel dissolution. its intensity increases as the cathodic potential increases. This result is related to an increase in deposition rate and corroborates our discussions during cathodic scanning. Such results have been widely reported in the literature (Abou-Krisha, 2007).

Electrodeposition of ZnNi Alloys Coating

In order to study the effect of both potential and Ni^{2+} ion concentration on ZnNi electroplating, cyclic voltammetry analysis was performed at various Ni^{2+} ion concentrations and potentials by applying the same parameters (V_b = 20 $mV.s^{-1}$, w = 250 rpm). The results were shown in Figure 2. As we can see on Figure 2, the voltammograms curves have a similar pattern, where we observe a significant evolution of both anodic and cathodic processes as a function of the imposed potential. Indeed, in all recorded voltammogram curves, the reduction current density increases as the applied potential increases. Also, the dissolution peak intensities showed a similar behaviour, i.e. the anodic peak intensity increased with rising cathodic potential. In addition, all plots show several peaks, suggesting the presence of several phases in the alloys as deposited during cathodic scanning. Such a result suggests that the chemical composition of alloys changes with cathodic potential as well as Ni^{2+} concentration. The presence of such peaks confirms the simultaneous co-deposition of zinc and nickel. It is important to note that the anodic peak for pur zinc dissolution was higher compared to the ZnNi alloy dissolution (Fig.1.a). Several hypotheses can be proposed for this behaviour. It may be due to the small amount of zinc deposited in the alloy compared with pur metal, or it may be attributed to the adsorption phenomenon, which remains competitive between different electroactive species (Zn^{2+} and Ni^{2+}). It can also be associated to the formation of some phases with nickel (Shams, 2018) Based on these results, it is clear that formation of ZnNi alloys with different compositions is achievable in our experimental conditions. It is also important to note that the shape of anodic peaks strongly changes with Ni^{2+} ion concentration.

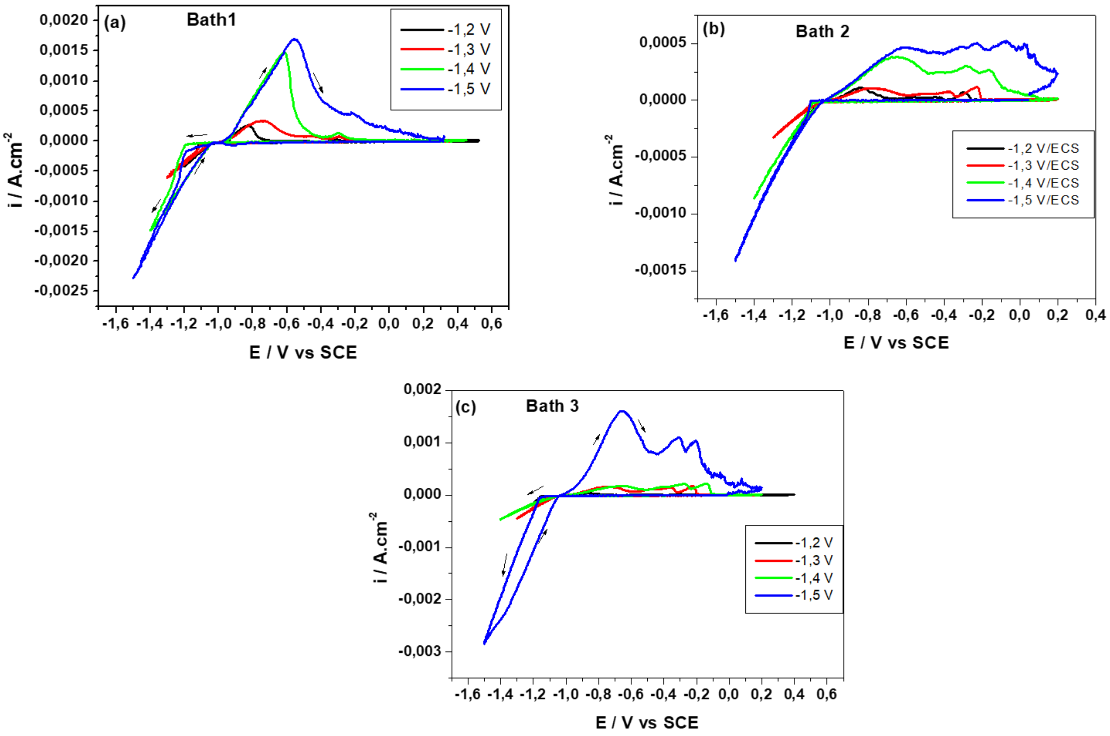

Figure 2. Cyclic voltammograms for ZnNi alloys deposition on vitreous carbon RDE at various Ni^{2+} concentration and cathodic limit potentials, (a) $[Ni^{2+}]$ = , (b) $[Ni^{2+}]$ = , (c) $[Ni^{2+}]$ = , pH = 2, Vb = 20 mV/s, w = 250 rpm

In the case of bath 1 with a concentration ratio ($\frac{[Ni^{2+}]}{[Zn^{2}]}$ = 0.66), we can see the emergence of a quite broad peak that is more pronounced around -1 V. It is attributed to zinc-rich phase compared with the kinetic study discussed of each element separately. In bath 2 (concentration ratio = 1), anodic peaks obtained at different potentials are flattened and not very symmetrical. They are attributed to ZnNi phases dissolution. As a result, their intensity remains similar and comparable, this may be due to the coexistence of several ZnNi phases with similar compositions. In such conditions, it is possible to consider the formation of the δ-ZnNi and γ-ZnNi phases, which has already been reported in the literature (Ghaziof, 2014)

For bath 3, with a concentration ratio of 2 , for potentials (-1,2, -1,3 et -1,4 vs. SCE), the anodic peaks are not changing with the imposed potentials. In this case, all peaks have a very flattened shape and low intensity, indicating the dissolution of different ZnNi phases. However, at the more cathodic potential (-1.5 V vs. SCE), a quite symmetrical anodic peaks were observed on the voltammogram. The first one is observed around the pure zinc potential and is more intense than the others. It is therefore attributed to the dissolution of a zinc-rich phase. Two other peaks, with low intensity compared to the first one, are located respectively at -0.42 and -0.27 V vs. SCE, which are quite close to the pure nickel dissolution potential. Obviously, these peaks can be attributed to ZnNi phases dissolution, which are probably rich in nickel.

Also, as we can see on the voltammogram, the peak intensities corresponding to zinc-rich phases are quite high compared with those of Ni-rich phases. This behaviour can be explained by the anomalous co-deposition phenomena, which means that the less noble metal is deposited preferentially compared to the noble metal. This phenomena has been widely reported and discussed in several research works related to ZnNi electroplating (Abou-Krisha,2007).

Current Efficiency of ZnNi Deposit

Based on the results as previously described, we can estimate the current efficiency of ZnNi deposited in each bath as a function of the applied cathodic potentials (figure 3). As shown in Figure 3, ZnNi current efficiency show almost identical evolution in all baths. In fact, deposition current efficiency increases as the potential becomes more cathodic. As a result, hydrogen evolution seems to be minimized under these conditions. These results clearly demonstrate the behaviour observed in the previously described voltammetric analysis, where a significant increase in anodic peak intensity was observed in all registered voltammograms. It is important to note that the current efficiency is strongly dependent on the Ni^{2+} ion concentration; the highest efficiencies were obtained for coatings produced from bath 2. In summary, ZnNi coatings obtained in different baths at highly cathodic potentials displayed a very considerable current efficiencies with an average value estimated at 80.8%.

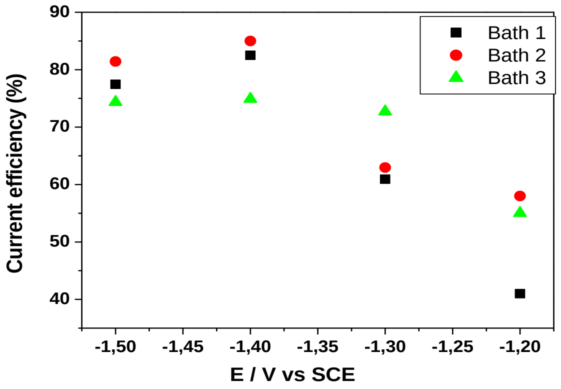

Figure 3. ZnNi current efficiency vs cathodic limit potentieals and different Ni^{2+} concentrations bath 1-[Ni^{2+}] = 0,2 mol/l , bath 2- [Ni^{2+}] = 0,3 mol/l , bath 3 - [Ni^{2+}] = 0,45 mol/l

Chronoamperometric Analysis

The obtained results from the previous sequence have allowed us to define the deposition parameters of ZnNi system and to confirm that ZnNi alloy deposition is possible in our experimental conditions. In this section, we will elaborate a series of ZnNi samples on steel substrates, from different baths (bath 1, bath 2, bath 3) with respectively Ni^{2+}/Zn^{2+} concentration ratios (0.66, 1 and 1.5) at -1.3V and -1,4 V vs. SCE during 600 s. The chronoammograms shown in figure 4 have almost the same shape and similar evolution. However it should be noted that measured current density changes with both potential and Ni^{2+} ion concentration. Indeed, current density in increased with increasing cathodic potential. This can be attributed to an increase of deposition rate, which probably induces a significant increase in the amount of deposit. It is important to note that the recorded current densities in bath 2 are much higher than those obtained from other baths. This correlates perfectly with our previous hypotheses as described from voltammetric analysis.

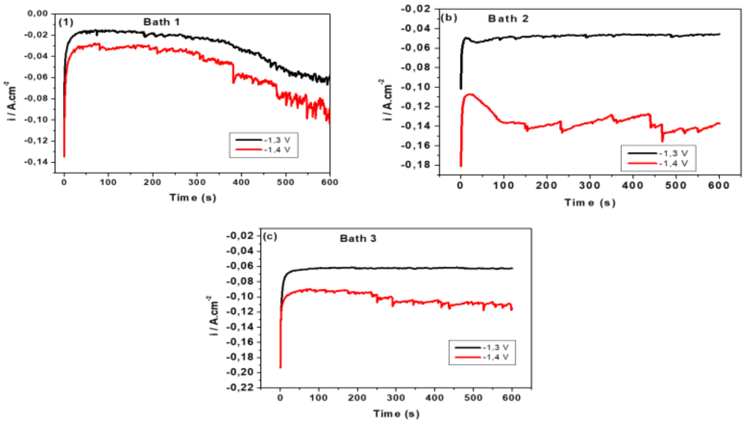

Figure 4. Current–time transients for ZnNi alloys coating deposited on steel substrate in différents baths and at various imposed potentials, pH = 2, t = 600 s, (a) bath 1, (b) bath 2, (c) bath 3

Surface Morphology of ZnNi Alloy Coatings

SEM micrographs of ZnNi alloy coatings produced from different baths at -1.3 V vs. SCE during 600 s are illustrated in Figure 5. As can be seen on Figure 5, the surface morphology of ZnNi coatings is stronbly dependend on the Ni^{2+} ion concentration in the electroplating bath. In the case of bath 1 (Figure 5.a), where the Ni(II) concentration is lower than that of zinc, the observed morphology is granular, and the coating consists of uniformly distributed grains as a background and some cauliflower-like microstructures growing on the surface of the deposit. According to some authors, the appearance of the cauliflower like morphology in the deposit suggests the formation of a nickel-rich ZnNi phase In addition, the presence of certain cavities (hollow shapes) in the deposit can be attributed to specific preferential sites of hydrogen evolution (Fashu, 2015)

Deposits formed from bath 2 with a concentration ratio = 1 (figure 5.b) , have a similar morphological aspect compared to bath 1 (figure 5.a), but there is a difference in grain size. In fact, the deposit exhibits a strong coalescence between grains, which have a tendency to form very fine cauliflower-like microstructures, which is uniformly distributed on the substrate surface. In this case, the coating is very compact with excellent coverage. In addition, some clear regions are uniformly distributed on the surface of deposit, this suggests the formation of different ZnNi phases with similar proportions.

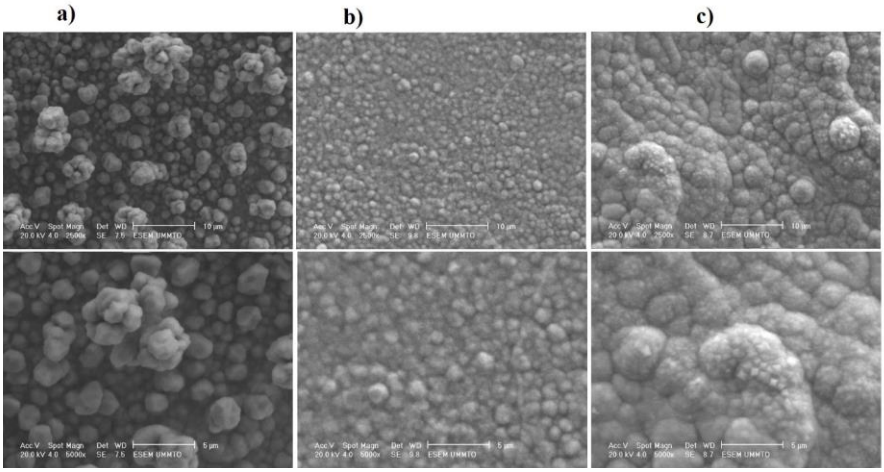

Figure 5. SEM Images of ZnNi alloys coating deposited on steel substrate at different Ni^{2+}/Zn^{2+} ion ratio in the bath , (a) bath 1 (ratio = 0,66), (b) bath 2 (ratio = 1), (c) bath 3 (ratio =) E = 1,3V vs SCE, pH = 2, t = 600 s

In the case of deposits obtained from bath 3 (concentration ratio = 1.5) figure 5.c, we can observe that the morphology is quite different from that observed in other baths (figure 5.a and figure 5.b). It consist of a fine pyramid-grains morphology. Globally, the deposit is compact but less homogeneous, with some areas highly agglomerated forming bumps with different sizes, which confers to the deposit a heterogeneous aspect. Such behavior can be explained by adsorption phenomena, which becomes competitive between H^+ and Ni^{2+} ions, leading a discontinuity in the deposit. Also, the presence of certain cracks in the coating is due to hydrogen evolution, which seems to be important in these conditions.

EDX Chemical Composition of Coating

In this work, we also performed a chemical composition analysis of ZnNi alloy coatings prepared in different baths at -1.3 vs. SCE. Figure 6 shows the obtained EDX spectra. As we can see on figure 6, all spectra show a characteristic peak of zinc and nickel. This confirms the simultaneous co-deposition of both zinc and nickel during ZnNi alloy coating electroplating. However, we note a difference in the peak intensities. In fact, all the recorded spectra show that the peak of zinc is more intense than the one of nickel, suggesting a high content of Zn in the deposits. Such behavior is probably explained by the anomalous co-deposition phenomena, which is widely discussed in the literature (Abou-Krisha, 2007). In this case, zinc is preferentially deposited compared to nickel. Other peaks with very low intensities are also observed, namely oxygen and carbon peaks. In this sequence, a semi-quantitative chemical composition analysis was also carried out on all deposits described above (Table 2).

It is clear that ZnNi deposits obtained at different Ni^{2+} ion concentrations have a high content of zinc. It should be noted that the content of each element evolves according to Ni^{2+} ions concentration. As a result, increasing the Ni(II) concentration in the bath induces a significant increase in Ni content and a slight rise in zinc content in the deposit.

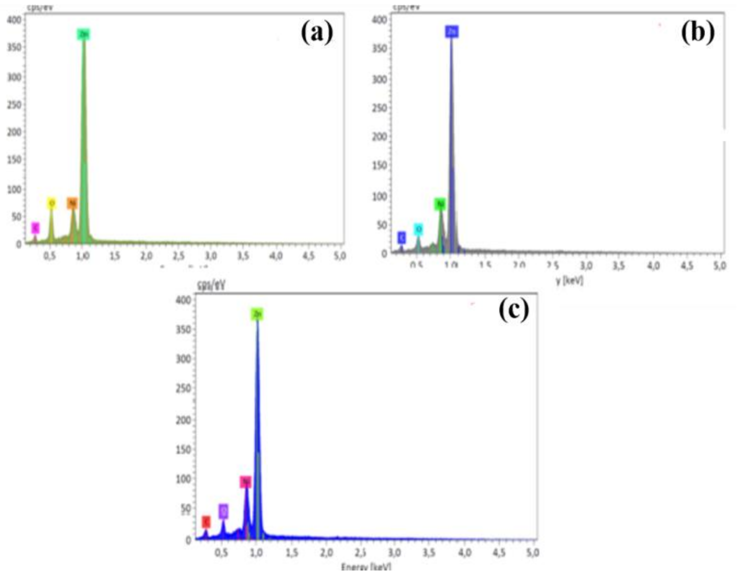

Figure 6. EDX spectra of ZnNi coatings prepared in different baths at -1,3 V vs SCE during 600 s, (a) bath 1, (b) bath 2, (c) bath 3

Table 2. Chemical compositions of ZnNi alloy coatings obtained by EDX analysis,
E = -1,3 V vs SCE, t = 600 s

Element content (wt.%)	Zn	Ni	O
Bath 1	78.49	10.11	11,4
Bath 2	80.78	13.45	5,77
Bath 3	78.47	17.36	4,17

It is clear that ZnNi deposits obtained at different Ni^{2+} ion concentrations have a high content of zinc (Table 2). It should be noted that the content of each element evolves according to Ni^{2+} ions concentration. As a result, increasing the Ni(II) concentration in the bath induces a significant increase in Ni content and a slight rise in zinc content in the deposit.

Corrosion Behaviour of ZnNi Alloy Coatings

In this section, we are interested to investigate the corrosion behaviour of ZnNi coatings in a 3 wt.% NaCl solution.

Open Circuit Potential Measurements

Figure 7 shows ocp curves of steel and ZnNi-coated steel obtained from different baths at -1.3 V Vs.SCE. These measurements were performed in an aerated 3% wt NaCl solution during one hour's immersion. As can be seen on Figure 7 , the corrosion potential of coatings is more cathodic compared to the one of steel. This confirms the sacrificial" character of the coating. Also, the plots reveal that the corrosion potential shifts towards noble values as the Ni^{2+} ions concentration increases. This could be due to the increase of Ni content in the deposit. It is important to note that these results are widely reported in the literature (Pedroza, 2014).

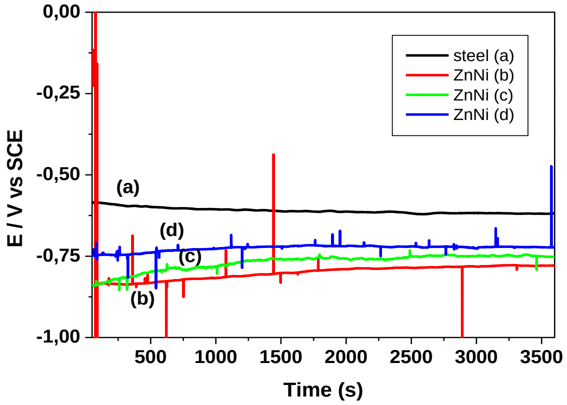

Figure 7. OCP curves of steel and ZnNi alloy coatings prepared from different baths at -1.3 V Vs.SCE after one hour's immersion in a 3% wt. NaCl solution.

Potentiodynamic Polarization Analysis

In order to highlight the electrochemical parameters which characterize corrosion behaviour of ZnNi alloy coatings, we carried out potentiodynamic polarization measurements of ZnNi alloys and steel in a 3% wt. NaCl medium, sweeping the potential of ± 250 mV around corrosion potential at a scan rate of 1 mV/s. The obtained curves are illustrated in figure 8. It is clear that the corrosion behavior of ZnNi alloy coatings is highly dependent on the Ni^{2+} concentration in the bath. it should be noted that the corrosion potential shifted to more positive value as Ni^{2+} ions concentration increased in the electrolytic bath Figure 8 (b, c and d). This can be explained by an increase of Ni content in the deposit, which corroborates the results of EDX analysis. The values of E_{corr} , i_{Corr}, b_a and b_c deduced from the previous curves (Figure 8) are shown in Table 3

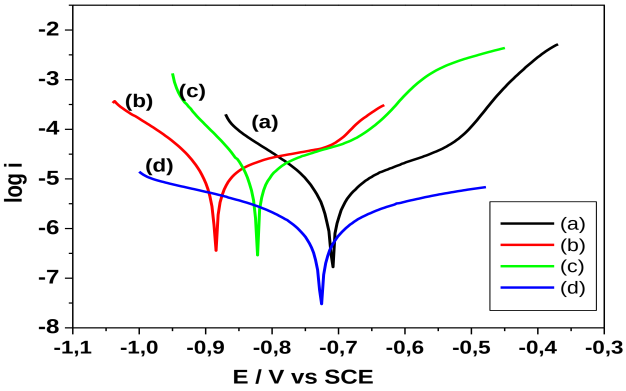

Figure 8. Potentiodynamic polarization curves for the teel and ZnNi alloy coating obtained in diffrent baths at -1,3 V vs. SCE, (a) steel, (b) ZnNi-bath 1, (c) ZnNi-bath2, (d) ZnNi-bath 3, after one hour's immersion in 3% wt. NaCl solution, $V_{b\,=}$ 1 mV/s

Table 3. Electrochemical parameters determined from potentiodynamic polarization curves (Fig.8)

Sample	$E_{deposit}$ = -1,3 V vs.SCE			
	E_{corr} / V vs SCE	i_{corr} (A/cm^2)	ba (mV/dec)	bc (mV/dec)
Steel	-0.700	$4.57 \text{ x}10^{-6}$	202	-101
ZnNi-bath1	-0.886	$1.81 \text{ x } 10^{-5}$	406	-118
ZnNi-bath 2	-0.822	$1.44 \text{ x } 10^{-5}$	237	-70
ZnNi-bath 3	-0.725	$1.50 \text{ x } 10^{-6}$	392	-321

From the values reported in Table 3, it can be seen that corrosion current density of ZnNi coating decreases as the concentration of Ni^{2+} increases in the bath, which confirms a further increase of Ni content in the deposits. Coatings elaborated in baths 1 and 2 exhibit higher corrosion current densities compared to the pure steel, this suggests that coatings are less resistant than steel, which lends them a protective character as sacrificial anodes.

It is also noticeable from table 3 that the coating prepared in bath 3 has a low current density, which certainly reflects its high corrosion resistance, because it contains a higher Ni content (around 17%) compared to the other coatings. In the light of these results, it is clear that corrosion behaviour of ZnNi alloy coatings is highly dependent on their chemical composition, which can easily be controlled by modifying the metal ion concentration ratio.

Conclusion

In summary, the electrodeposition of ZnNi alloy coatings is quite feasible in our experimental conditions. Nevertheless, the deposition kinetics revealed a strong dependence on potential and ions concentration ratio. Indeed, both valtammogram processes (anodic and cathodic) evolve as a function of the imposed potential and Ni^{2+} concentration. In this case, the corresponding current densities increase with cathodic limit potential. In addition, voltammetric analysis has shown that the shape of anodic peaks changes considerably according to the ions concentration ratio, which due to the formation of several ZnNi phases. It has been revealed that ZnNi coatings obtained from batch 2 exhibited a better morphological aspect compared to those obtained from other baths. The EDX chemical analysis indicated that ZnNi coatings obtained in different baths have a high zinc content and low Ni content, which indicates that the electrodeposition of ZnNi is affected by the anomalous codeposition phenomena. The study of the corrosion behaviour of coatings developed at various Ni^{2+} concentrations has clearly confirmed their protective character as sacrificial anodes. In addition, Increasing Ni^{2+} ions concentration in the bath improved corrosion resistance of ZnNi alloys coating.

Scientific Ethics Declaration

The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

Acknowledgements or Notes

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