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A Case Study of the Wastewater Treatment Methods for Alkaline Zinc-Nickel

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ABSTRACT

The alkaline electroplating solutions for plating zinc-nickel alloy coatings are a specified coating in use for automotive and other critical applications requiring high corrosion resistance versus traditional zinc deposits. The composition of the alkaline zinc-nickel solution has chelating properties and potential by-products of complex cyanides entering process rinse waste streams, batch systems or combined wastewater treatment systems. The ability to meet environmental compliance requirements for metals zinc, nickel, and for chemical and biological oxygen demand (COD or BOD) provide challenges for applicators using these systems. An overview of current wastewater treatment methods and options for enhanced treatment is discussed, including the benefits of organic oxidation in the presence of iron species, as well as the use of proprietary metal precipitation chemistry.

Introduction

Zinc coatings have a history over many decades to provide corrosion protection in many types of air and water environments, including high pollution areas to all types of parts, components and fastener systems. Since the 1960 period, zinc coating systems have also evolved to provide improved corrosion performance. These deposits primarily resulted from alkaline and acid electroplating systems providing increased corrosion performance as shown in Fig. 1. Further performance improvements were realized with new conversion coating systems and top-coat development, but it was not until zinc alloy technology was introduced that demonstrated a much higher degree of improvement for corrosion resistance performance.

The protective value of the zinc plating is partially a result of its ability to form a protective layer (by products formation) during the process of corrosion of the zinc surface when applied over ferrous substrates, such as carbon steel. In this zinc-steel combination, the mechanism of protection from the zinc coating is an anodic electro-potential. This sacrificial tendency, while the zinc layer corrodes preferentially to protect the steel from attack, can be referenced by the number of hours of exposure in a simulated salt fog environment, referenced in ASTM B117 test procedure. During the corrosion process, as the zinc layer is degraded, white corrosion salts are formed on the surface, and once that layer is fully penetrated, the steel-substrate red corrosion will begin to occur, commonly referred to as red rust.

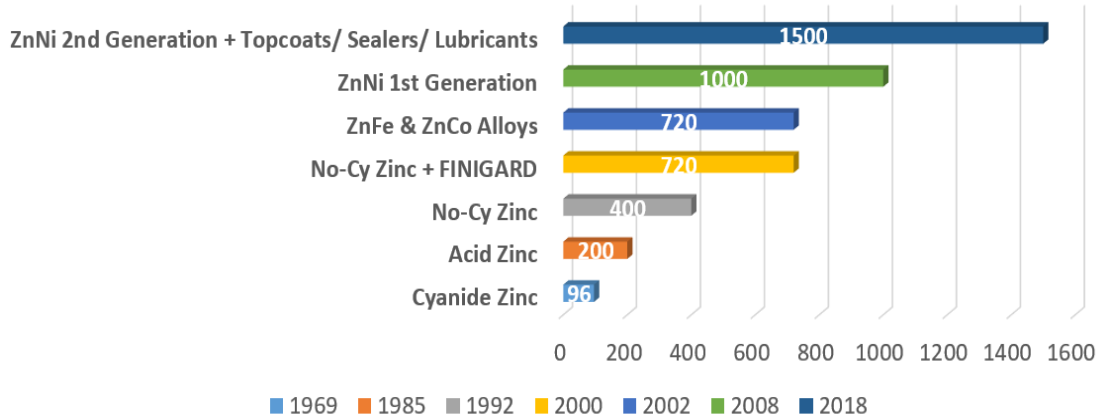
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Figure 1: Trend NSST Hrs Red Corrosion:
Year Evolution & Type Zinc Deposit



Pure zinc coatings provide a degree of protection for the substrate based on type of pure zinc, layer thickness and conversion coating type. However, in a severe corrosion environment, zinc is consumed more rapidly and the protective value becomes much reduced. Attempts in the past, by simply increasing the zinc deposit layer thickness, were not commercially cost effective. While the thicker coating often caused problems during post plating forming or welding of components, it was higher electroplated thickness ranges from plating which had more deposit failures, including poor adhesion and blistering that reduced the protective value. With increasing quality demands, higher performance coatings have been a target of development over many decades.

Consequently, over the past 20 years, most industries - especially the automotive, industrial equipment and building sectors - began looking for alternative processes that could offer higher performance, originating from demands for higher quality requirements for meeting extended warranty programs, and less claims for failures.

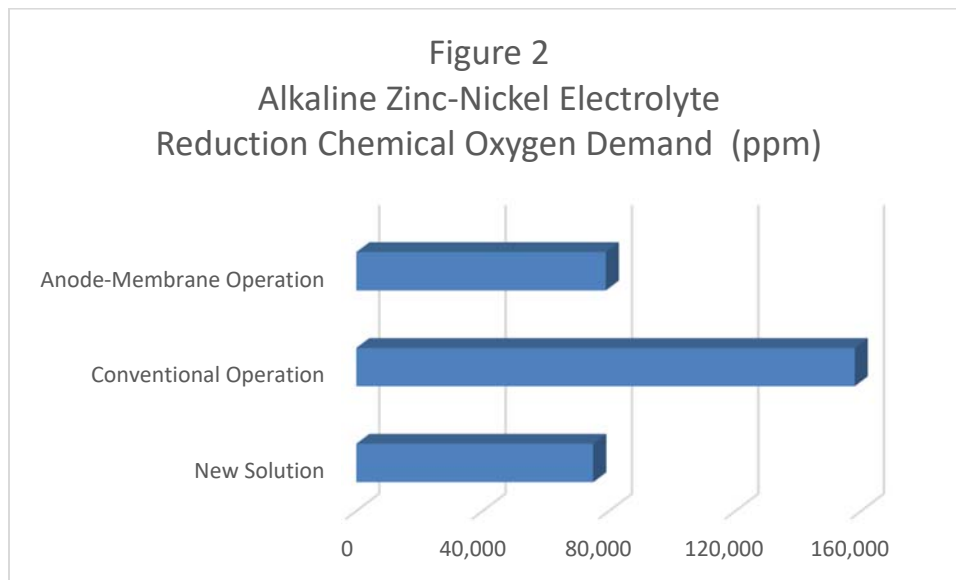
The Surface Finishing Industry answered the call for improved performance through the introduction of viable commercial binary zinc alloy deposits. These systems included zinc-Iron, zinc-Cobalt and zinc-nickel alloy deposits that dramatically reduced the corrosion rate of the zinc-alloy plated layer. The boosted performance of these coatings, while maintaining 0.0002 to 0.0005-inch (roughly 5 to 12-micron) deposit thickness ranges, provided the different OEM companies and engineers viable options toward increasing corrosion performance further with zinc-nickel alloys. These first generation chemistry systems, based on alkaline electrolytes similar to non-cyanide zincate solutions for pure zinc, provided zinc-nickel alloys ranging from 5 to 16 wt% nickel deposits. These second-generation alkaline zinc-nickel systems, utilizing specific types of alkaline amines in their formulations, allow for depositing 12 to 16 wt% nickel that results in gamma phase alloys. These offer corrosion performance superior to all other types of zinc alloy deposits. However, they create more difficulties in the waste management side of processing for applicators who utilize these technologies.

Today an alkaline electrolyte, depositing a gamma phase 12 to 16 wt% zinc-nickel alloy, can achieve up to 480 hr of resistance to white (zinc) corrosion and up to 1500 hr, sometimes more to base metal (ferrous substrate) attack with 0.0005 inches (12 microns) as shown in Fig. 1. These systems offer the OEM companies and engineers many additional deposit performance advantages over other zinc or zinc-alloy deposits. However, applicators who are tasked with providing these coatings now face challenges with existing waste treatment protocols, resulting from these electrolytes, demonstrating a stronger chelation constant from their design, and electroplating by-products formation during the deposition process.

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Evolution of zinc-nickel

The second-generation electrolytes also can incorporate the use of proprietary membrane anode technology.¹ Overall, the use of this technology offers advantages for alkaline zinc-nickel electrolytes, while also considering the wastewater treatment plan. Their use changes the wastewater characteristics, realizing decreases in demand for oxidation and reductions with the complexing strength of the solution. Figure 2 demonstrates the results for Chemical Oxygen Demand (COD), comparing a new alkaline zinc-nickel solution electrolyte makeup compared to six months of operation as a conventional system versus operation of the same electrolyte with a membrane anode system.



With the membrane anode technology, there is essentially zero-to-insignificant generation of cyanide complexes, which have a higher affinity for complexing zinc and nickel, entering the rinse water system. Overall, the organic loading of the zinc-nickel plating solution creates difficulty for the oxidation of the complexing amines derived from the process rinse water. In this case, organic loading includes total organic carbon (TOC), Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD) and carbonate concentrations. The membrane anode technology reduces the TOC loading in the rinse waters up to 40 - 60%, which is important in the treatment plan and efficiency of the treatment process. This is demonstrated in the data.

However, despite an anode membrane system, the composition of the alkaline zinc-nickel solution with process rinses continues to have strong chelating properties at entry into either a batch or combined wastewater treatment system that must be accounted for today. Regardless of the operating improvements, incorporating alkaline zinc-nickel electroplating solutions into your facility does require the need and consideration for an improved water treatment program.

Waste treatment challenge

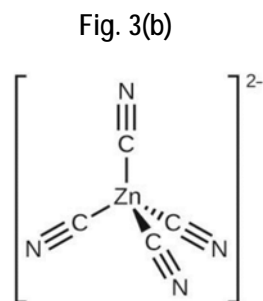
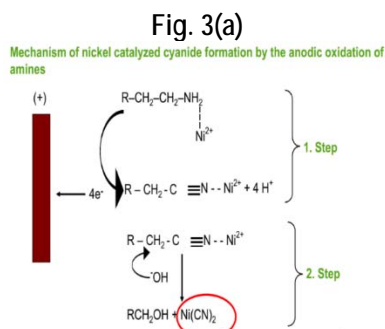
The ability to meet local environmental compliance, in parts per million (ppm) or parts per billion (ppb) concentrations of zinc and nickel, challenges applicators using alkaline zinc-nickel technology, either with or without a 3S membrane anode system¹ technology.

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Due to the strong complexing nature of these electrolytes using alkaline amines in their design, and as required for making zinc-nickel deposits, natural occurring formation of by-products, including sodium carbonates, is common. As carbonates from the zinc-nickel-plating solutions increase in the waste stream rinses, zinc and nickel solubility increase and the process for precipitating these metals as “sulfides” becomes less efficient. As listed in Table 1, “Theoretical Solubility in mg/L, Selected Metals in Pure Water (25°C)”, the use of metal precipitants in the water treatment that form ‘metal sulfides’ will assist in meeting effluent requirements.

Table 1: Theoretical Solubility mg/L; Selected Metals in Pure Water (25°C)			
Metal	As Hydroxide	As Sulfide	As Carbonate
Cadmium (Cd ⁺²)	2.3E-05	6.7E-10	1.0E-04
Chromium (Cr ⁺³)	8.4E-04	No ppt	NA
Cobalt (Co ⁺²)	2.2E-01	1.0E-08	NA
Copper (Cu ⁺²)	2.2E-02	5.8E-18	NA
Iron (Fe ⁺²)	8.9E-01	3.4E-05	NA
Lead (Pb ⁺²)	2.1E+00	3.8E-09	7.0E-03
Manganese (Mn ⁺²)	1.2E+00	2.1E-03	NA
Mercury (Hg ⁺²)	3.9E-04	9.0E-20	3.9E-02
Nickel (Ni ⁺²)	6.9E-03	6.9E-08	1.9E-01
Silver (Ag ⁺¹)	1.33E+01	7.40E-12	2.10E-01
Tin (Sn ⁺²)	1.1E-04	3.8E-08	NA
Zinc (Zn ⁺²)	1.1E+00	2.3E-07	7.0E-04

Applications without the membrane anode system technology generate cyanide complexes that further complicate any considerations for a treatment protocol plan. Suppliers of these zinc-nickel technologies have similar characteristics of greater chelation potential of solution resulting from the options available for the organic amines utilized in the plating system that break down during electrolysis. This complex formation, as shown in Fig. 3(a and b), provides challenges for meeting compliance parameters including BOD and COD demands, and for zinc or nickel metal limits.



Cyanide from the conventional alkaline zinc-nickel process acts as a strong chelant to zinc and nickel. The complexed metal-cyanide formation is shown as example for zinc.

Traditional wastewater treatment alkaline zinc-nickel

Observations globally indicate the current treatment method for rinses is not “waste” source segregation, but instead combining of waste sources in a common “neutralization tank” and applying the wastewater treatment methods. The

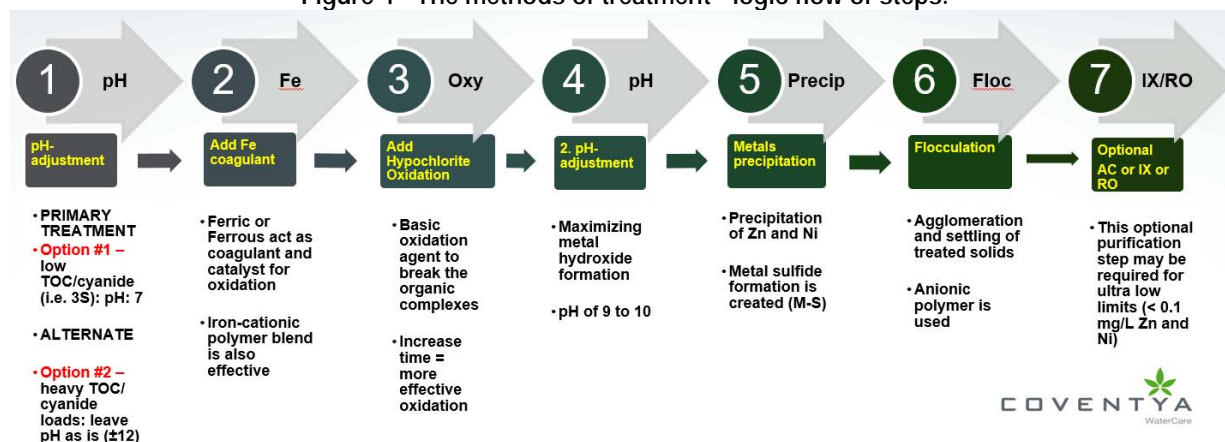
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rationale here is that the combined overall plating wastewater may adequately dilute the complexing chemistry coming from the rinsing. This scheme, however, only works **IF** a large dilution is realized from other plant wastewater sources; **THEN** the treatment burden is reduced. In these cases, large dosages of heavy metal precipitant are commonly applied, and a final effluent polishing with activated carbon and/or a final ion exchange polishing media is often the case. Given this, what are the best methods of design for treating alkaline zinc-nickel wastewater?

Advanced wastewater treatment plans

Segregation of alkaline zinc-nickel rinses from the combined wastewater flow is "*the best standard practice*" for providing a viable and effective treatment. Figure 4 outlines the logical sequence flow for a defined method of treatment following with an explanation of each step and the significance for each step.

Figure 4 - The methods of treatment - logic flow of steps.



Step 1: Initial pH adjustment. This step is the initial pH adjustment of the process wastewater stream. The pH 7 approach is derived for conditions where there is lower overall TOC loading. Alternatively, where there are higher TOC loadings, and where there is cyanide by-product from not using a membrane anode system, a higher pH of 12 is the proper treatment protocol. Figures 5 and 6 (below) provide a greater detailed overview of each option.

Step 2: Coagulation with iron. The use of ferrous or ferric iron species have both found success. Our research has identified the use of a ferric/cationic polymer blend that will create enhanced solids agglomeration and settling with improved water clarity. Lower final effluent COD values are more readily obtained in this case.

- The use of iron species with oxidative precipitation of metallic ions, including organic molecular oxidation and entrapment, is well documented in the literature for the purposes of environmental remediation.³
- Iron in use with oxidation is effective. Our research identifies the use of ferric chloride (Fe^{+3}) and/or ferrous chloride (Fe^{+2}) as effective when used as the catalyst/coagulant in the treatment process. It is proposed that the use of ferrous iron species may, in some cases, provide for enhanced metal removal and organic entrapment due to the iron valence change during the oxidation process with hypochlorite.
- Use of spent pickle acid as an iron source is an option for applicators who generate a volume of spent acid from the surface prep activation or other plant operations.

Step 3: Oxidation: The use of 12 wt% sodium hypochlorite is effective in breaking the complexes. The study includes demands of 2.5, 5 and 10 vol% hypochlorite amounts to gauge effectiveness.

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- The use of iron species and hypochlorite oxidation for the reduction of BOD and COD was studied and documented in the literature.⁴ In both cited references, the mechanisms of co-precipitation / oxidation of complexing organics / adsorption of neutralized solids are reviewed and discussed.
- Hypochlorite oxidation can be carried out at neutral and elevated pH ranges for meeting the treatment requirements of alkaline zinc-nickel rinse waters. The most commonly used treatment pH value for alkaline zinc-nickel rinse waters is 7. Hypochlorite oxidation in the presence of iron creates hydroxyl radical formation. This hydroxyl formation is very effective in organic destruction (amines, other process organics).

Steps 4 and 5: pH adjust & sulfide precipitation. Using metal precipitants will reduce the nickel and zinc concentrations through sulfide precipitation. The use of a carboxamide formula (1) is used for the nickel sulfide (Ni-S) reaction, while a mineral sulfide formula (2) works for the zinc (Zn-S) reaction. The use of a proprietary blend can also be considered for the precipitation of both zinc and nickel.

- Various metal precipitants (MP) are known to be used to assist in the wastewater hydroxide precipitation process in efforts of reducing the final metallic concentrations by forming M-S (metal sulfide) species, in the wastewater treatment process.²
- These MP products are very effective for reducing zinc and nickel concentrations to the required compliance concentrations. Our research indicates selective metal precipitants have affinities for zinc and nickel in this process after the oxidation sequence is completed. Metal precipitants of the carboxamide group of molecules have an affinity for nickel precipitation forming (Ni-S) nickel sulfides. Metal precipitants of the sulfide-mineral group have an affinity for zinc precipitation forming (Zn-S) zinc sulfides. A manageable wastewater operation should target to <1 ppm for Zn and Ni removal in most countries. In the Asian marketplace, compliance requirements are observed as low as 0.1 ppm for nickel. It is possible to utilize a formulation combining the two metal precipitants into one reagent. We used a proprietary Coventya water treatment formulation of this composition for our case study.¹

Step 6: Flocculation: The agglomeration and settling of the treated solids is assisted with the addition of anionic polymers with about a 40% charge density.

- **Proprietary cationic polymer/iron blends.** Our research identifies the benefit use of a Coventya product to further agglomerate suspected solids in the treatment process, thereby eliminating solids carryover and improving wastewater clarity in the final effluent stream. Lower COD can be realized by the use of an iron / cationic polymer blend.

Step 7: Ion exchange, reverse osmosis or activated carbon / clay can be considered viable in a final stage as necessary to further improve the quality of the water leaving the plant/facility, especially if local regulations are very stringent toward metals concentration. In the Asian marketplace, it is common to observe a final batch polishing with activated carbon or clay derivatives, such as locally sourced coconut shell carbon. In these applications, a secondary flocculation of combined mixed carbon/clay effluent is applied as a final tertiary step.

Case study examples

Overall, looking at the optimization of treatment efficiency, the process zinc-nickel rinsewater metal loading should be less than 400 mg/L for zinc, and less than 70 mg/L for nickel. This represents approximately a 2 to 3 vol% loss of solution as drag-out of the electroplating electrolyte and is a normal expectation in most installations. This rinsewater segregation into a batch treatment tank is acceptable for smaller treatment volumes. However, applicators with a much higher flow rate can design a “flow-through” tank-sizing model for meeting the retention times required for the proper oxidation and metal precipitation reactions.

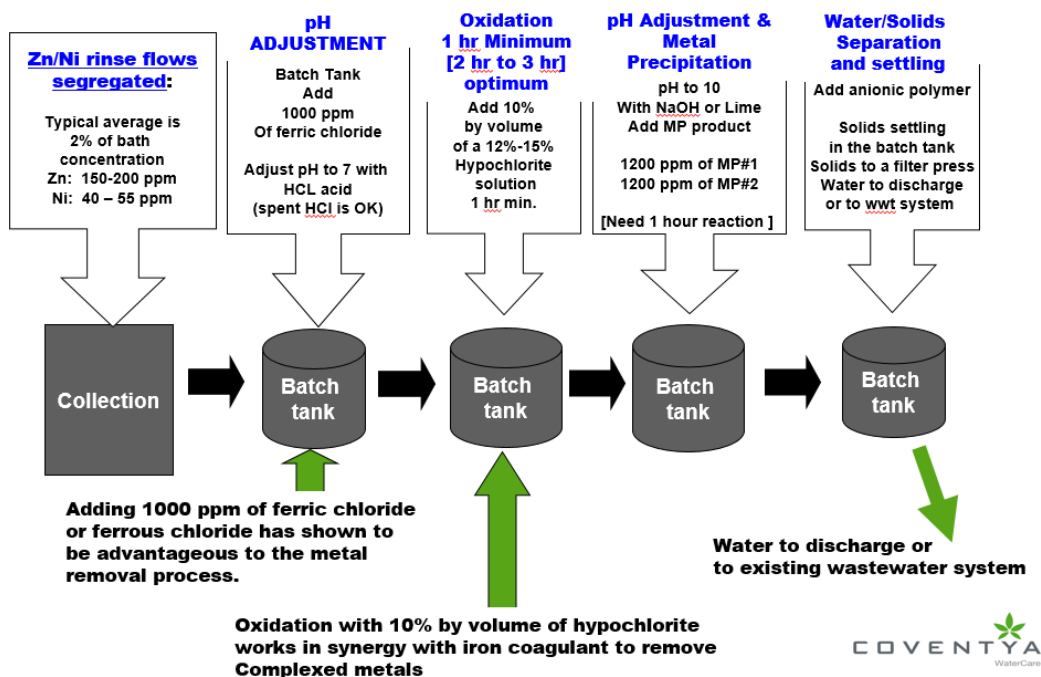
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Where membrane anode technology is utilized in the alkaline zinc-nickel plating process, the total organic loading (as TOC) is reduced, less carbonate formation is realized, and the cyanide (CN) formation is eliminated, simplifying the treatment process. Because of these reduced wastewater characteristics, the overall process treatment demands for organic amine oxidation and zinc and nickel metal complexing are more effective. Two options, in Figs 5 and 6, represent treatment details that have been proven effective for meeting effluent requirements from zinc-nickel rinsewaters. The details are provided for each process option considering the initial selection of pH or any adjustment considerations.

In these two figures, each option that summarize the methodology of treatment for providing a predictable model for efficient removal of metallic zinc and nickel, also including a reduction of BOD/COD are overviewed.

Treatment Option (1) in Fig. 5 shows the details for the steps utilizing an anode membrane system in the electroplating electrolyte. Here, the oxidation with hypochlorite at a pH of 7 is effective in the organic destruction of zinc-nickel rinse solutions with the characteristics of lower TOC and lower cyanide formation. In these solutions, the use of hypochlorite at this pH provides for maximum amine fractioning and oxidation.

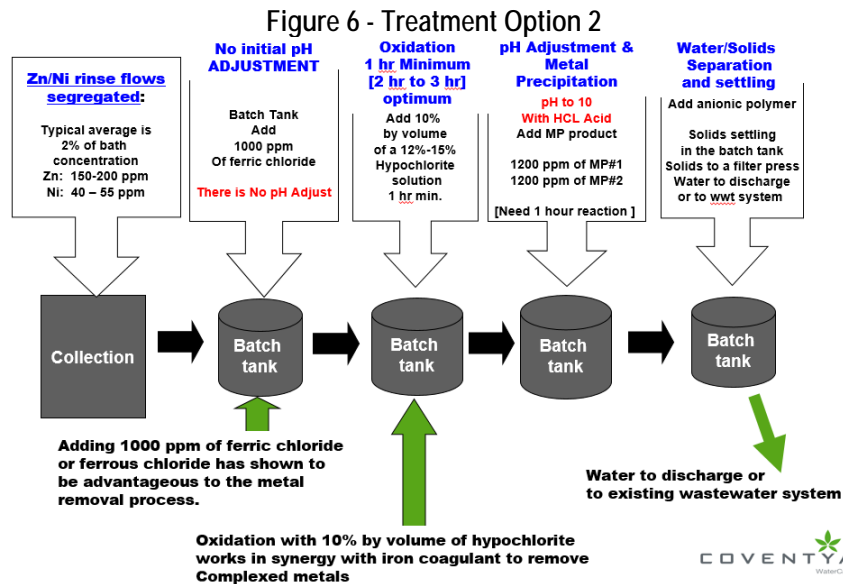
Figure 5 - Treatment Option 1



Treatment Option (2) in Fig. 6 overviews the details for the steps considering a zinc-nickel electrolyte without using a membrane anode system where higher TOC and concentrations of complex-cyanide are more likely to be the result.

The oxidation with hypochlorite at a pH level of 12, as shown in Fig. 6 is effective in organic destruction of zinc-nickel rinse solutions with the characteristics of the higher TOC and higher cyanide formation. This allows for improved destruction of the cyanide complex degrading from cyanide to cyanate, and then to elemental C–N–OH. Our research indicates a 12 wt% solution of sodium hypochlorite is the best vehicle of solution oxidation. Other oxidants such as H₂O₂ / ozone are not as effective as sodium hypochlorite. A minimum of 1 hr is required for oxidation. Improved higher efficiency reductions can be realized if more time is allocated for the treatment.

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Results of treatment methods

The following Tables 2, 3 and 4 represent a predictive model of metal removal at oxidation levels using a 12% sodium hypochlorite solution at dosage levels of 2.5, 5 and 10 vol% originating from collected rinsewaters from a zinc-nickel plating process. The reaction time was 1 hour. Comparisons are made in the case of a conventional zinc-nickel operation with traditional anodes, versus a zinc-nickel operated with a membrane anode system.

Table 2, for Treatment Option #1, summarizes the results for the metals precipitation where the zinc and nickel concentration (mg/L) represented in the rinse stream was adjusted to a pH of 7.0. In all treatment schemes, the coagulant used was an iron-cationic polymer blend at 1000 ppm, and metal precipitant, blended at 2400 ppm.

Rinsewater metals concentration by Zn-Ni operation Type	Table 2: Treatment for metals				
	Rinse concentration pH adjusted to 7.0		12 wt% hypochlorite dilution		
			2.5 vol%	5 vol%	10 vol%
Conventional Operation	Zinc	298 mg/L	8.7 mg/L	5.1 mg/L	1.8 mg/L
	Nickel	48 mg/L	2.3 mg/L	1.2 mg/L	0.8 mg/L
Membrane Anode Operation	Zinc	173 mg/L	0.7 mg/L	1.0 mg/L	0.3 mg/L
	Nickel	32 mg/L	1.8 mg/L	0.1 mg/L	0.04 mg/L

As shown, using Treatment Option #1 for both the conventional and membrane anode operation, metal concentrations for zinc and nickel decrease based upon the concentration of hypochlorite added for oxidation. A 10% v/v of hypochlorite provides the maximum metal removal. For the membrane operation, lower overall metal removal is observed because of the reduced overall organic loading.

Table 3, for Treatment Option #2, summarizes the results for the metals precipitation including the cyanide oxidation from a conventional zinc-nickel process. Here the zinc and nickel concentration (mg/L) and cyanide (mg/L) represented in the rinse stream was adjusted to a pH of 12.0, a requirement for the cyanide being present in the rinse

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water stream. The reaction time was 1 hour. In this treatment scheme, as in the Table 2 data, the coagulant used was an iron-cationic polymer blend at 1000 ppm and metal precipitant (MP), blended at 2400 ppm.

Rinsewater Cyanide, Zn and Ni Concentration	Table 3: Treatment for cyanide and metals				
	Rinse concentration pH adjusted to 12.0		12 wt% hypochlorite dilution		
			2.5 vol%	5 vol%	10 vol%
Zn:Ni Conventional Operation	Zinc	378 mg/L	0.7 mg/L	1.0 mg/L	0.3 mg/L
	Nickel	59 mg/L	1.8 mg/L	0.1 mg/L	0.04 mg/L
	Cyanide	3.5 mg/L	0.5 mg/L	0.5 mg/L	0.4 mg/L

As shown, using treatment Option #2, metal concentrations for zinc and nickel decrease, based on the hypochlorite concentration added for oxidation. 10 vol% of the concentrated hypochlorite provides the maximum metal removal with minimal cyanide residual remaining after treatment.

Table 4 for the Organic Balance provides a summary of treatment results for oxidation of organics from a conventional operation of a zinc-nickel process rinse utilizing regular anodes, compared to operation with a membrane anode system. The rinse stream pH was adjusted to 7.0. The reaction time was 1 hour. In this treatment scheme, as with the Table 2 data, the coagulant used was an iron-cationic polymer blend at 1000 ppm, and metal precipitant (MP), blended at 2400 ppm.

Rinse Water Organic Loading Concentrations by Zn-Ni Operation	Table 4: Treatment for organics				
	Rinse Concentration pH adjust to 7.0		12 wt% hypochlorite dilution		
			2.5 vol%	5 vol%	10 vol%
Conventional Operation	TOC	1350 ppm	920 ppm	257 ppm	116 ppm
	BOD	75 ppm	619 ppm	282 ppm	117 ppm
	COD	2900 ppm	663 ppm	420 ppm	111 ppm
Membrane Anode Operation	TOC	320 ppm	380 ppm	332 ppm	194 ppm
	BOD	80 ppm	110 ppm	619 ppm	165 ppm
	COD	1100 ppm	995 ppm	135 ppm	88 ppm

As demonstrated, the starting organic concentrations of TOC/COD are significantly lower in the rinses from the membrane operation vs the conventional operation. For the conventional operation, up to 10 vol% of the hypochlorite for oxidation reduces the concentrations of TOC / BOD / COD as shown. For the membrane operation, with lower TOC / BOD, the range of hypochlorite oxidation does little to demonstrate reductions, but for the COD, up to 10 vol% hypochlorite does show significant reduction. Using the membrane anode operation still could use less hypochlorite at 5 vol% for savings in treatment.

Overall summary

In the case study work, some best practices, focused on the treatment of process wastewaters originating from alkaline zinc-nickel electroplating solutions, can be realized for those systems that operate a membrane anode system, or those with a conventional anode system. Some conclusions of the work include the following:



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- Zinc-nickel process rinsewaters should be segregated from other plant waste treatment sources and separately treated for providing viable and effective treatment. This approach improves the efficiency of the process for reduction in metal concentrations, BOD, COD and cyanide.
- Our research indicates that two (2) options of treatment can be utilized, based on the pH, with respect to TOC and the presence of higher concentrations of cyanide. Option #1 is oxidation at a pH 7, while Option #2 is oxidation at pH 12, due to the elevated TOC/cyanide concentrations in the waste stream.
- Oxidation with hypochlorite and with an iron catalyst present, from using spent acid or ferric or ferrous addition, is effective at breaking the types of complexes encountered in alkaline zinc-nickel solutions. A minimum one-hour retention time is required, with more time yielding improved treatment results, for further reduced COD, BOD, zinc and nickel metal.
- Metal precipitation chemistry, after oxidation, is effective in meeting regulatory compliance limits for zinc and nickel.
- A membrane anode process for the zinc-nickel plating system produces a waste stream profile, with lower TOC, no cyanide and lower carbonate concentrations, which is easier to oxidize at a much lower hypochlorite concentrations (a range of 2.5% to 5.0% is common vs 10% for conventional). The lower TOC also creates reduced final waste sludge by a factor of up to 50% vs. the conventional treatment at higher pH values.
- A proprietary coagulant based on an iron-cationic polymer blend and a proprietary metal precipitant blend is effective in improving the treatment efficiency of zinc-nickel process rinsewaters.

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About the authors



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He holds a Bachelor of Science in Chemistry, with 36 years of Industry experience including technical, marketing, research and executive management that includes industry presentations and written publications for the plating and surface finishing industry. For more than two decades, he has been an active member of Committees *B08, *F07 & *G01 for American Society for Testing and Materials (ASTM) International. He is a member of Society of Automotive Engineering International (SAE), National Association of Corrosion Engineers (NACE) and has been a member of the Electroless Nickel (EN) Steering Committee, additionally serving as Technical Director for the 2007 and 2010 EN Conference Symposia.

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*ASTM B08, Metallic and Inorganic Coatings, *ASTM F07, Aerospace and Aircraft & *ASTM G01, Corrosion of Metals.