



Removal of Zinc from Barrel Plating Wash Water by Electro Coagulation using Aluminium as Anode and Stainless Steel as Cathode

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ABSTRACT— Heavy metals are generally refractory and cannot be de-toxified and de-graded biologically, hence it is a challenging task for the industrialist and environmentalist for the safe method for the effective disposal of waste containing heavy metals. Various technique which have been developed for the removal of heavy metal include chemical precipitation, reverse osmosis, bio-sorption etc. The electro coagulation method was widely used and playing an important role in the effluent treatment. Since electro coagulation process has better removal efficiency compared to chemical coagulation. The current topic is aimed for the removal of zinc as Zn^{2+} . The main objective to the current work is to remove zinc from barrel plating wash water by electro coagulation using aluminium (Al) as anode and stainless steel (SS) as cathode. The scheme study involves: Effect of initial metal concentration (866ppm, 433ppm, 216.5ppm, 279ppm), Effect of pH (acid, alkali, neutral), Effect of current density (0.1A, 0.2A, 0.6A), Effect of supporting electrolyte (NaCl) concentration (2gpl, 4gpl), Effect of polarity change (AL-AL, MS-MS), Effect of different combination of electrodes (AL-SS, MS-SS) for the removal of zinc. The crystallographic orientation, identification of structure were analyzed by X-ray diffraction. The presence of different phases was analyzed by XPS analysis. In order to study the surface morphology and composition of coatings SEM and EDAX were used. The reduction of zinc in wash water has been determined by FT-IR spectrometer and HPLC. In particular the electro chemical techniques provide higher efficiency than conventional methods.

Keywords— Electroplating, Electro coagulation, Zinc, Barrel Plating.

INTRODUCTION

Environment is viewed with different angles by different environmentalists. It is the sum of all social, economical, biological, physical (or) chemical factors which constructed the surroundings of man, who is both creator and molder of his environment. Fortunately, however, environmental awareness also grown dramatically, especially in the past few years. Several nations around the world are taking the lead in the implementation of new laws that regulate, and in many cases even ban, the use and disposal of hazardous chemicals. Among various types of pollution, water pollution is of major criteria which require immediate attention. This is because life is dependent on water crucially.



Water pollution is the introduction of contaminants into fresh or ocean waters of chemical, physical, or biological material that de-grades the quality of the water and affects the organisms living in it. This process ranges from simple addition of dissolved or suspended solids to discharge of the most insidious and persistent toxic pollutants (such as pesticides, heavy metals, and non-degradable, bioaccumulative & chemical compounds). Depending upon the nature of contaminates in the effluent and relative quantities of these effluents from different operations, the extent of water pollution potential of any wastewater is characterized by the following factors colour and odour, suspended solids, dissolved solids, pH, acidity, alkalinity, hardness (temporary and permanent), Biological oxygen demand (BOD), Chemical oxygen demand (COD) and toxic anions and cations. Electro plating industry is one of the major industries for water pollution. Because, in Electro plating industry water is a main source for kiering, dyeing etc., the effluent from dyestuff manufacturing industries is generally highly colored with large amount of suspended solids and organic contents. Discharge of this wastewater into water bodies is objectionable not only in aesthetic consideration but also due to the fact that it consideration but also prevents re-oxygenation and reduces the penetration of sunlight through the body of water. Electro plating dyeing and finishing industry is one of the major polluters among industrial sectors. In Electroplating industry, as the name implies base metals are plated for protection from corrosion for brightness, and for fair appearance. The plating is usually done by using chromium, nickel, zinc, cadmium, copper, silver and gold etc. The operations are produced by cleaning and stripping which contribute strong effluents in significant quantities.

Barrel plating can accommodate a wide variety of shapes and sizes as well as for different metals and alloys. These parts can then be plated in an equally diverse amount of plating baths each with their own properties and formulations. Generally any part that can fit through the opening in the barrel can be plated provided good procedures are maintained, excepting long & thin parts. The quality of the finish can equal that of still or rack plating and is usually more economical. Typically parts weighing 5 or less or having a volume of 125 cu. in. or less can be routinely barrel plated. The rotation of the barrel and the parts within it lend themselves to other advantages. The mechanical energy of the rotation produces a burnishing action that helps to clean and descale the parts to a greater degree than rack plating, much the same as wringing your hands under the water faucet facilitates the speedier removal of soils and dirt from them. The tumbling action is also responsible for the high degree of plating uniformity which can be achieved in the inner surface of barrel. As the parts move they make and break electrical contact creating what is called a bi-polar effect whose net benefit is the leaving out of the high and low areas which could pose enormous problems if the same parts were to be racked and finished. The primary function of barrel plating is to provide an economical means to electro plate manufactured parts that also meets the customer's specific finishing requirements. Conventionally wastewater from plating industries was treated for the following methods: 1. Physical treatment, 2. Biological treatment, 3. Chemical and electro chemical treatment. Various electrochemical methods are classified as follows, electro chemical oxidation, electro reduction, electro deposition, electro dialysis, electro floatation, electro sterilization, electro coagulation, electro filtration, electrophoresis and electro fenton.

When the particulates size fall into the sub sieve range and do not settle down, it becomes necessary to transform the same into larger aggregates by coagulation and then allow them to form sediment for separation. The most commonly used coagulants are aluminium sulphate, ferric sulphate, ferrous sulphate, chlorinated coppers, and ammonia alum or potash alum and sodium aluminate and long chain organic compounds called polyelectrolytes. They have charged functional groups, which may be anionic or cationic in nature. Inorganic coagulants have a wide spread use and have the disadvantage of contributing substantially to the sludge volume, organic flocculants are specific in use and effective at low dosages.

Electro coagulation refers here to the electrochemical production of destabilization agent



that brings about charge neutralization for pollutant removal. Electro coagulation is simple and efficient method where the flocculating agent is generally made of iron or aluminium. In this process, the treatment is done without adding any chemical coagulant or flocculants thus by reducing the amount of sludge, which must be disposed. The electro coagulation has been successfully used to treat oil wastes, with a removal efficiency as high as 99%. A similar success was obtained when treating dye-containing solution potable water, urban and restaurant wastewater and water containing nitrate or fluoride.

Experimental Material and Methods: Composition of Barrel Plating Wash Water (Zinc Sulphate Bath: Zinc Sulphate (0.20M), Sulphuric acid (0.01M), Sodium sulphate (0.4M), Boric acid (0.16M), Anode (graphite) & pH (2.75). The following Instruments are used for the experimental setup (fig 1), D.C .Power supply with ammeter and voltmeter, magnetic stirrer, spectra lab (speed), aluminum (anode), mild steel (anode) & stainless steel (cathode).

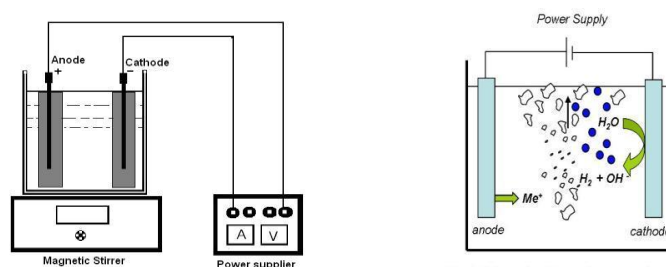


Fig. 1: Schematic of basic electrocoagulation process

Fig. 1 Schematic diagram of Electro-chemical cell

EXPERIMENTAL DETAILS: The electrode used in this study consists of aluminium plates and stainless steel plates of 99.99% purity. All the chemicals were of analytical grade and the reagents were prepared using double distilled water. A raw (stock) solution containing zinc wash water of 866 mg/L was used in the experiment. The working samples consist of 100% 50% and 25% of solution prepared by dilution with distilled water to required levels. The samples were used freshly from the ice cold deep freezer by dilution from the stock as and when required. Before the experimentation, the pH of the solution was maintained to be 6.3. Other pH conditions i.e., 6.0 7.0 and 8.0 were obtained by using 0.1N H₂SO₄ and 0.1N sodium hydroxide solution. All measurements were carried out at ambient temperature. The removal of zinc in wash water in terms of current density, pH, supporting electrolyte variation were determined by electro coagulation. From the results percentage reduction of zinc was calculated. Spectral analysis of the samples was carried out using FT-IR, HPLC and SEM/EDAX.

Results and Discussion: When electro coagulation is carried out using SS (cathode) Al (anode) at constant current density of 0.6 A/dm² effective removal was observed for lower concentration followed by 50% and to the lowest zinc removal percentage was observed at 100% initial concentration(C₀).The extent of zinc removal is represented as 25%>50%>100%. The observed voltage was found to be decrease from 3.1V to 2.7V in 15 minutes and exponential increase during next 15 minutes in case of 100% concentration. Similarly a constant voltage of 2.7V was observed during first 5 minutes and rest of 25 minutes respectively. In case of 25% the interesting part is that at 50% of initial concentration the voltage was 2.4V and 2.2V upto 5 minutes and rest of the period. It can be observed that the amount of current consumed is visibly lower at 50% than other concentrations i.e., 50% dilution is best suited for removal of zinc at a reduced current consumption. Therefore, at higher C₀, the amount of hydroxide produced was not enough to remove all the zinc molecules causing a decrease in the zinc removal efficiency. In addition, at higher C₀, Al(OH)₃ that forms



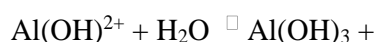
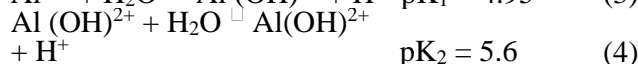
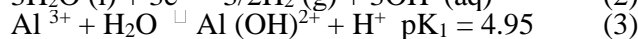
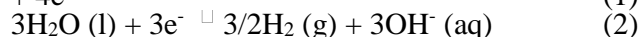
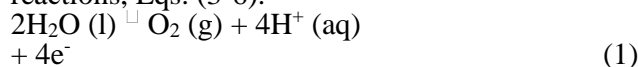
near the surface fouls the electrodes and intermediate products form in the solution, which block the active site of the electrode. Both of these factors decrease the zinc removal efficiency. With an increase in C_0 , the resistance increases, this leads to higher power consumption. It is quite clear that under the present experimental conditions, lower zinc concentration results in better zinc removal efficiency.

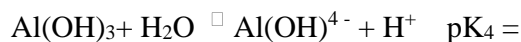
Under the experimental conditions of current density being varied from 0.1 to 0.6 A/cm², solution pH of 8.0. The highest current (0.6 A/cm²) produced the quickest removal rate. This expected behavior is easily explained by the increase of coagulant and bubbles generation rate, resulting in a more efficient and faster removal, when the current is increased. Indeed, the amount of aluminium hydroxide ions generated within the electro coagulation cell at a given time are related to the current flow using faraday's law. Although the current density was increased from 0.1 - 0.6 A/dm² zinc removal efficiency is increased. The amount of zinc removal depend on the quantity of adsorbent (Aluminium hydroxide) generated, which was related to the time and current density. Hence, the amount of zinc adsorption increased with an increase in adsorbent concentration, which indicates that the adsorption depend on availability of binding sites for zinc.

Here maximum removal was observed at both alkaline and neutral condition than acidic conditions. The main reason is that at alkaline condition OH⁻ are readily available and at neutral conditions also OH⁻ are generated instantly. Inducing a congenial environment for effective electro coagulation. However, there is some edge observed for zinc removal at neutral condition compare to alkaline condition. Because the fresh unreacted OH⁻ ions are generated, where as alkaline condition the available OH⁻ ions would have developed ionic bond with other ionic pollutants to some extent. As mentioned earlier, the zinc removal at both alkaline and neutral condition was observed to be effective with an edge at neutral condition. Surprisingly, zinc removal in acidic medium achieved 100% removal within 30 minutes this may be due to the H⁺ ions would have not favoured electro coagulation of pollutants to take place during 15 minutes. However very fast rate zinc removal was observed in the second half of 15 minutes. The OH⁻ ions generated would have exceeded the H⁺ ions at some point of time, reached a point above which supplest OH⁻ ions would have continued to a quick removal of dissolved zinc. The resulting voltage is observed to be lower for neutral condition compare to acidic and alkaline conditions. At 25% maximum removal was observed at acidic followed by alkaline and neutral conditions. This may be due to that at a lower dilution the electro chemical reaction would have been faster probably, because of faster ionic movements compare to higher concentrations.

So, the process of insitu generation of OH⁻ ions and its mobility would have been faster at acidic condition. The voltage was observed to be lowest at acidic condition 5th minute onwards, whereas it is observed to be constant between 5-7pH. The results revealed that the removal efficiency was maximum for acidic < neutral < alkaline, the pH was found to increase during the EC process, this equilibrium pH represents a dynamic balance between the complex chemical reactions involving a H⁺ or OH⁻ donor and acceptor during EC. The outcome of the stable alkaline pH can be attributed to the cathodic water reduction, Eq. (2), being predominant over the anodic water oxidation, Eq. (1) and aluminium hydroxylation

reactions, Eqs. (3-6).





5.6 (6)

At $\text{pH} < 6$, the protons in the solution are reduced to H_2 , and thus, the proportion of hydroxide ion formation is less. Consequently, zinc removal efficiency is lowered. Precipitation and adsorption are the two major interaction mechanisms considered during the EC process. Each of the mechanisms is applicable at different pH ranges. At low pH values, metal species, e.g., Al^{3+} , generated at the anode bind to the anionic site of the zinc molecules, thus neutralizing their charge and reducing their solubility. This process of removal is termed as precipitation. The adsorption mechanism operates at higher pH ranges ($\text{pH} > 6$) and involves adsorption of organic substances on amorphous metal hydroxide precipitates.

At 100% C_0 the zinc was removed within 15 minutes (2g of NaCl). The lower amount of supporting electrolyte will remove the zinc effectively. At increasing the amount of supporting electrolyte the removal efficiency is low. The voltage was increased with increasing the amount of electrolyte. As mentioned above in 50% C_0 the zinc was completely removed at 30 minutes (2g of NaCl). The voltage was increased with increased amount of electrolyte.

At 25% C_0 the zinc removed at 15 minutes onwards (2g of NaCl). Consequently, lower C_0 concentration and at lower the amount of supporting electrolyte, the efficiency was effective. As discussed earlier the voltage was raised with the increase in supporting electrolyte.

The addition of sodium chloride is necessary to increase the electrical conductivity of the waste water to a suitable level. Power consumed is directly proportional to the cell voltage and increases with fluid resistivity. Sodium chloride was selected as an electrolyte because of its low toxicity at moderate levels, its reasonable cost, and also the chloride species avoids inhibition phenomena at the surface of sacrificial anodes. However, an excessive concentration of sodium chloride was shown to be detrimental to process efficiency. An excessive amount of sodium chloride induces over consumption of the aluminium electrodes, and dissolution becomes irregular. At NaCl concentrations above 3 g/l, current yields of Al dissolution were higher than 150%.

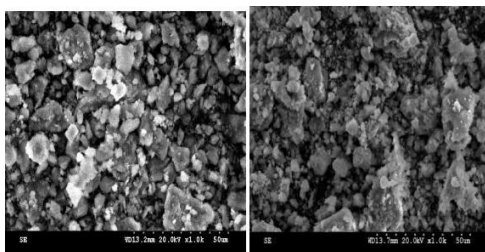


Fig2. SEM image of sludge (a) Al-Al electrode (b) Fe-Fe electrode

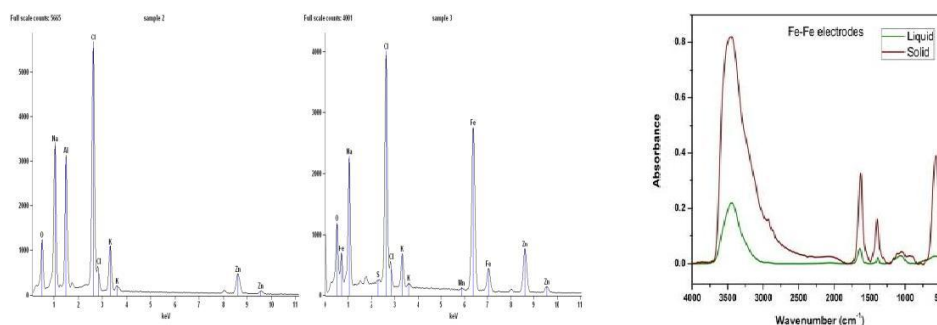


Fig.3 EDAX spectrum of sludge a)Al-Al b)Fe-Fe

Using Al as anode: The extent of zinc removal from barrel plating wash water in alkaline medium is higher compare to acidic medium for the reasons already explained. Although alkaline medium is given a higher removal rate, the composition of effluents (OH^- , Cl^- and other anions) has favoured the alkaline medium than the acidic medium for electro coagulation with reference to zinc. At constant voltage of 1.3 it is observed throughout for both acidic and alkaline medium.

Using MS is anode: The extent of zinc removal is higher in acidic medium than in alkaline medium. Perhaps due to the physical dissolution of mild steel into its ionic form (Fe^{3+}).

The microstructure of the films has been observed by scanning electron microscopy (SEM) method. Fig.2 (a & b). presents the morphology of the sludge shows an aggregated formation of oxide particles with an average grain size of about 15-20 μm . Fig.2(a & b) shows the presence of fine coagulant particles on the surface. The average grain size was observed to be 10-15 μm . The SEM images clearly suggest the agglomeration of iron precipitate in the presence of Zinc.

EDAX analysis was used to analyse the elemental composition of the sludge. The weight percentage and atomic percentage of the sludge (Al-Al electrode) compositions are given in Table.1 & Fig 3a. Shows the presence of O, Na, Al, Cl, K and Zn elements, respectively. An EDAX spectrum of a sludge (Fe-Fe electrode) shows, the presence of the O, Na, S, Cl, K, Mn, Fe and Zn elements, respectively and the values are given in Table.2 & Fig 3b Composition of sludge (Fe-Fe electrode) as determined using EDAX analysis. The Fig.4(a&b) shows the FT-IR spectrum of sludge (Al-Al electrodes) and liquid. The sharp and strong peak at 3442.8 cm^{-1} was due to the O-H stretching vibration. The minor peak at 2091 cm^{-1} indicates the allenes stretching. The strong peak at 1639.83 cm^{-1} was assigned to H-O-H bending. The peaks at 1318.04 , 1070.86 , 743 and 620.52 cm^{-1} were assigned to Sec alcohols, Primary Alcohols, Ar, HC respectively.

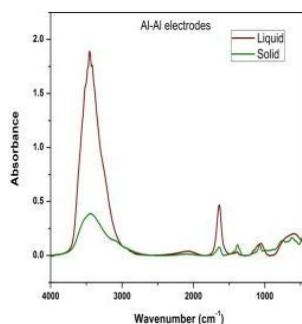


Fig.4FTIR spectrum of sludge a) Al-Al b)Fe-Fe



FT-IR spectrum of Fe-electrode of sludge and liquid.(Fe-Fe electrode) showed, OH stretching, alkenes and stretching, H-O-H bending, sec alcohols, primary alcohols, Ar HC and Cx at 3452, 1638, 926 and 620 cm^{-1} respectively. Fig 5(a,b,c). Shows the retention time vs concentration profile on HPLC response for various plating effluents. In this analysis, the efficiency for the electrochemical treatment is found as high as 99% shows the complete removal of zinc ions (as zinc chloride). The ion electrode shows the maximum removal than aluminium electrode.

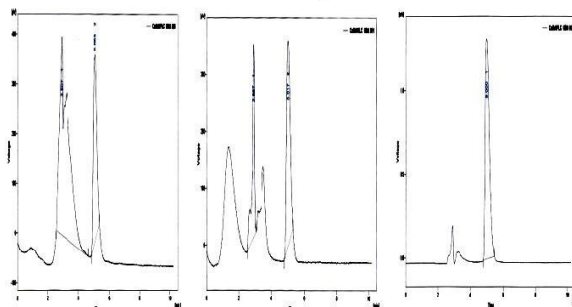


Fig.5 HPLC analysis of sludge a) wash water b) Al-Al post EC c) MS-MS post EC

CONCLUSIONS

The results of the present study show the applicability of electro coagulation in the treatment of barrel plating wash water containing zinc. This study generated important information on the optimal conditions of several variables that influence electro coagulation. The lower Zn initial concentration results in better Zn removal efficiency. The higher current density produced the quickest treatment with an effective reduction of zinc in the barrel plating wash water. The more effective removal capacity was reached at the pH range between 8-10. The addition of NaCl is often necessary to increase the electrical conductivity of the wash water to a suitable level. An excessive amount of NaCl induces over consumption of the aluminium electrodes and dissolution becomes irregular. The frequent change of electrode-polarity may provide an efficient way for removal of both organic and metallic pollutants from water. HPLC, FTIR for understanding the extent of pollutant removal with reference to organic composition. The sharp and strong peak at 3442.8 and 3452 cm^{-1} was due to the O-H stretching vibration. In HPLC analysis, the efficiency for the electrochemical treatment is found as high as 99.9% shows the complete removal of zinc ions (as zinc chloride). Using Al as anode the zinc removal efficiency is higher in alkali than acidic where as in the case MS as anode the zinc removal efficiency is higher in acidic than alkali medium. SEM image shows an aggregated formation of oxide and fine coagulant particles. EDAX spectrum shows the presence of other elements in the sludge.

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