

**Electrochemical Treatment of Wastewater Containing Cyanide and Zinc**

المعالجة الكهروكيميائية للصرف الصناعي المحتوى على السيانيد والزنك

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يهدف هذا البحث لدراسة تجريبية مبنكرة لعملية كهروكيميائية لإزالة السيانيد والزنك فى آن واحد من الصرف الصناعى وقد تم اجراءالتحارب باستخدام خليه كهريه مزودة بقطب من الجرافيت المسامى وتم دراسة تأثير كثافة التيار ومعدل السريان الحجمى للمحلول وكذلك درجة القلوية على كفاءة الإزالة وكفاءة التيار وذلك من خلال ثلاث مجموعات من التجارب .

المجموعة الأولى تم اجراؤها على محلول يحتوى على السيانيد فقط لثلاث تركيزات مختلفه منه وقد وجد أن تركيز أيون السيانيد بعد عمليه المعالجة يتناقص مع زيادة كثافة التيار ومعدل السريان الحجمى وكذلك درجة القلوية للمحلول ولهذا السبب فإن كفاءة عملية الإزالة تزيد حتى تصل الى الحد الأقصى ثم تتناقص تدريجيا وذلك عند معدل سريان حجمى قدره ٢٥ مل/دقيقة ،وكلا من كفاءة الأزالة وكفاءة التيار بزيادة مع زيادة معدل السريان الحجمى وزيادة درجة القلوية للمحلول. وتم أيضا دراسة تشغيل الخلية فى الوضع الأقفى ووجد أن الوضع الرأسى يحقق نتائج أفضل من الوضع الأقفى.

والمجموعه الثانيه من التجارب تم تنفيذها على محلول يحتوى على الزنك فقط ووجد أن كفاءة ازالة أيون الزنك فى الخليه تزيد مع زيادة كثافه التيار ولكن كفاءة التيار تتناقص.

أما المجموعه الثالثه تم تنفيذها على محلول يحتوى على كلا من أيونات السيانيد و الزنك ووجد أن كفاءة عملية الأزالة للسايانيد وازنك تزيد مع زيادة معدل السريان الحجمى وزيادة كثافة التيار وزيادة تركيز الأيونات ووجد أيضا أن كفاءة الأزالة للسايانيد تزيد مع زيادة قاعديه المحلول وتظل دون تغير فى حالة الزنك . وكفاءة التيار لأزالة الزنك تزيد مع زيادة كلا من كثافة التيار ومعدل السريان وكذلك التركيز وتظل ثابتة مع درجة القلوية أما كفاءة التيار لأزالة السيانيد تتناقص مع زيادة كثافة التيار وتزيد مع زيادة معدل السريان الحجمى والتركيز وكذلك درجة القلوية للمحلول.

**ABSTRACT**

A study was carried out to investigate the feasibility of using a packed bed electrochemical reactor with flow-through porous graphite electrodes to remove either cyanide and/or zinc ions simultaneously from wastewater. The effects of cell current density, flow rate, pH and inlet concentration on removal efficiency and current efficiency of either cyanide ion or zinc ion and combination of both ions removal were studied. It was found that the removal efficiency of cyanide ion increases with cell current density, pH and feed flow rate. Several cell arrangements were investigated. It was found that the vertical arrangement of cell is better than the horizontal arrangement. For zinc removal it was found that removal efficiency of zinc ion increases with cell current density. For a combination of both ions it was found that removal efficiencies of zinc and cyanide increase with flow rate, current density and inlet concentration. Current efficiency of zinc removal increases with current, flow rate and zinc ion concentration but remains constant with pH change.

*Key words: Cyanide, zinc, electrochemical cell, flow-through porous electrodes, wastewater*

## 1. INTRODUCTION

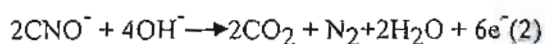
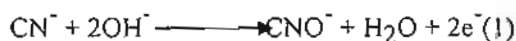
Metal plating has been identified as an environmentally risky industrial sector. The potential hazardous nature of the waste streams often contain high amounts of metals such as nickel, copper, zinc, chromium as well as cyanide [1,2]. Electroplated zinc coatings are widely used for the protection of cast iron and steel against corrosion. Alkali-cyanide baths are commonly used. Thus, cyanide complexes with zinc often occur in the plating rinsewater dispensed from electroplating plants. The effluent also contains acidic and alkaline rinses, spent baths and solutions, accidentally spilled liquids, and rinse waters [3,4]. Minimizing the environmental impact of the metal plating activity can best be achieved by the adoption of pollution prevention, pretreatment and cleaner production methods prior to final effluent treatment [5,6,7]. In addition, the remaining waste can be further treated by conventional methods such as chemical precipitation [8,9,10,11,12,13].

Cyanide and zinc ions are considered poisonous substances which pollute water and which need recovery, removal or destruction [14, 15]. Several treatment processes are available for cyanide removal using chlorine, ozone, and hypochlorite and hydrogen peroxide [15]. Cyanide can be treated by conversion to carbon dioxide and nitrogen through alkaline chlorination [4,16,17]. This process was carried out in two steps: First, cyanide is changed to cyanate at pH 10.5–11, using calcium hydroxide to raise the pH. The reaction requires 60 min for 100% completion. Second, chlorination at pH 8 was made to convert the cyanate into nitrogen and carbon dioxide gases. The latter reaction requires 1 h to complete. The complete conversion of cyanate was carried out by a careful reduction of pH-value from 11 to 8 in the presence of excess chlorine. Cyanide must be pretreated not only to meet disposal requirements, but also because it acts as a

complexing agent that inhibits precipitation [18, 19]. Zinc can also be deposited with ferric chloride, aluminum sulfate, powdered & granular activated carbon and by using chlorination used in conjunction with coagulation and filtration through sand filter [20].

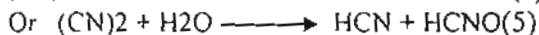
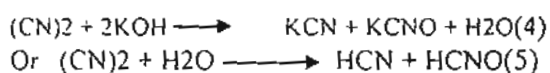
Electrochemical method obviates the use of dangerous chemicals and the formation of toxic volatile compounds containing chlorine when using alkaline chlorination. Chemical methods suffer the drawback of generating a large quantity of sludge, which contains heavy metal after chemical decomposition and the electrochemical method, in theory, has the advantage of decomposing cyanide at the anode and collecting heavy metal at the cathode simultaneously without generation of sludge [21]. Socha et al [22] study the electrochemical oxidation of dilute solutions containing cyanide complexes of zinc using a carbon felt anode. The composition of the solution was determined using stability constants for particular complexes. The application of a carbon felt electrode enables the oxidation of cyanides (10 mmol dm<sup>-3</sup>) in 99.2% at 0.7 V. Under these conditions, the electrical energy consumption is about 24 kWh. Modification of the anode surface with copper oxides allows a cyanide conversion degree of 99.6% at 0.7 V with electrical energy consumption of 17.6 kWh. In this article, a novel process is investigated in which both cyanide is oxidized and removed at the anode of an electrolyzer and zinc is deposited at the cathode.

Tamura et al [23] found that the mechanism of cyanide oxidation at the anode is as follows:

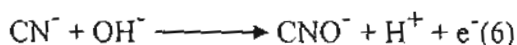


Hine et al [24] found that "with PbO<sub>2</sub> coated electrodes, the current density and temperature do not affect the current

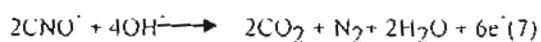
efficiency". However the current efficiency does drop significantly when the cyanide concentration is below 0.2 M. Tissot and Fragniere [25] found that the detailed mechanism of the reaction is still controversial, but it has been generally accepted that the first step of the electrochemical oxidation of cyanide is as shown previously in equation (1). This reaction is slow on platinum and graphite and on more active anodes like stainless steel, magnetite or lead. Electro deposited lead dioxide on titanium or stainless steel seems to be a good compromise between a good efficiency and acceptable corrosion resistance. Kuhn [26] said that in particular that cyanide was oxidized on platinum without oxygen evolution. There appear to be at least two reaction mechanism for its formation one being alkali catalyzed polymerization and the other being reaction of cyanogen with alkali or water as follows:



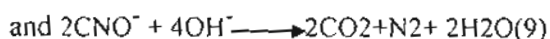
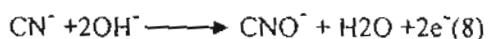
Sawer and Day[27] reported ammonia as an oxidation product again in terms of reactions of the cyanogen with the solution. The limited data of Dart et al[28] indicate no difference between results obtained on graphite and platinised titanium. Dodge et al [29]discussed an anodic reaction as follows:



Lure and Genkin [30] also state this reaction to occur, as well as the following reaction:



Easton [31] stated that the following two reactions take place, but Rumi and Topinka[32] found that cyanide converts into ammonia and carbonate



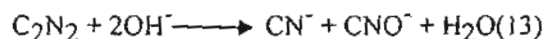
Christopher et al[33] discussed the system chemistry where the solution of cyanide and its electrochemical oxidation are rather complicated depending on the reaction conditions. The reaction products are observed but the primary reaction is converting the cyanide to the much less toxic product, namely, cyanate and the reaction that decreases current efficiency in excess of hydroxide with the evolution of oxygen.



The conversion of cyanide to cyanate exhibits large overpotential but it can be catalyzed by copper. Katagiri et al [34] showed that cuprous ion in a cyanide complex is oxidized anodically and the products are cuperous ion and cyanogen which is hydrolyzed to cyanate and that when the concentration of hydroxide becomes small the anodic reaction must change, in particular, cyanide is oxidized in the one electron process.



The cyanogen  $\text{C}_2\text{N}_2$  thus formed will react rapidly when it encounters hydroxide in solution according to the following reaction:



When the anolyte and catholyte are mixed, the pH will rise and any cyanogen appearing in the anolyte will be converted to cyanate. Lanza and Bertazzoli [35] reported that mechanism involving  $\text{Zn}^{2+}$ , Zn and  $\text{H}^+$  species is as follows:



## 2. EXPERIMENTAL

### 2.1. Materials

All solutions and suspensions were prepared from 17 M $\Omega$ /cm resistivity deionized water (Corning, MEGA-PURE System, MP-290, Corning, NY). The Preparation of synthetic solutions was carried out as follows:

#### 2.1.1. For cyanide ion

Cyanide solution was prepared by dissolving 2.5 g of potassium cyanide 99% purity, in distilled water until completely soluble. This solution was diluted to obtain different concentrations of cyanide. The experiments were carried out on three solutions:

(A) First solution containing 1 ppm of cyanide and having pH of 4.5, 5.5, 7, 9.6 and 10.

(B) Second solution containing 10 ppm cyanide and having pH of 4.5, 5.75, 7, 9.25 and 10.

(C) Third solution containing 100 cyanide and having pH of 3.09, 4.5, 7, 9.76 and 11.

#### 2.1.2. For Zinc ion

Zinc solution was prepared by dissolving of 1.0 gm of zinc metal 99% purity in a small amount of HNO<sub>3</sub> and boils the mixture in an electric heater to expel the red vapors of nitrogen oxides. then it was diluted with distilled water to give the desirable concentrations. The experiments were carried out on one solution containing 143 ppm of zinc ions.

#### 2.1.3. For both zinc and cyanide ions

The solution contains 2.0 ppm concentration of cyanide and also 5.4 ppm concentration of zinc with pH. of 2.4, 6.3 and 9.6. pH of solution was adjusted using

sulfuric acid and was measured by a pH meter.

### 2.2. Method

#### 2.2.1. Cell construction

A bench scale electrochemical cell was constructed to study the feasibility of simultaneous removal of cyanide and zinc and to give the designer information needed for the design of the industrial-scale cell. The cell is shown in Fig.1. It consists of two porous electrodes (stainless steel rod placed in graphite flakes) in a plexiglass cylinder. Each compartment was 16 cm in diameter and 12 cm high. The anode compartment was placed above the cathode compartment and the influent enters at the bottom of the cell.

Two plexiglass perforated disks were placed at each compartment to support the graphite flakes. The cell terminals consists of stainless steel rods 1 cm in diameter and 10 cm long which were connected to a DC power supply. Perfect tightness was necessary to prevent leakage and to ensure good electrical contact between electrodes and current collectors. This was achieved by compression using four external screws of tie rods and nuts located on the two plates and plexi-glass covered the two cell compartments. The treated effluent leaves through an opening at the top of the cell. The electrical circuit used in this work is shown in Fig (2). Potential and current are measured between cathode and anode. The cell was connected to a DC power supply conducted between anode and cathode.

Two types of power supplies were used throughout this work, one for potentials up to 30V and currents up to 3A of type (Manacor PS. 303) and the other consisted of home-made rectifier capable of supplying potentials up to 55V and currents up to 10A. Both cell potential and current were measured using digital multi-meter of type DTJ530B

### 2.2.2. Experimental procedure

1-Feed solution was fed to the cell by gravity from an overhead tank with controlled flow rate using needle valve.

2-Effluent samples from the anode compartment were collected, and were transferred into a dark refrigerator for 24 hours to prevent the decomposition of cyanide or zinc.

3-The current and potential of the cell were measured after reaching steady-state.

4- For each run, 16 samples of effluent stream were collected at ten minutes intervals and also 12 samples were collected every fifteen minutes afterwards.

5- Each run was stopped after reaching steady state conditions (when constant concentration for zinc or cyanide was obtained).

### 2.3. Measurements

All samples were diluted as needed by adding de-ionized water before analysis. CN<sup>-</sup> ions concentration was analyzed by colorimetry followed the pyridine-barbituric acid procedure as described in standard methods. The color was measured by using a UV-visible spectrophotometer (HP-8452A, Germany) at a wavelength of 578 nm. The zinc concentration in effluent streams was determined with Nanocolor meter the MACHEREY-NAGEL. GmbH & Co. KE Postfach 101352-D-52313 DUEREN

## 3. RESULTS AND DISCUSSIONS

### 3.1. Effect of operating conditions on the outlet concentration, removal efficiency and current efficiency of cyanide ion alone:

#### 3.1.1. Effect of current density

The effect of current density (the ratio between cell current and the geometric cross sectional area of the cell) on the outlet

concentration of cyanide is shown in Fig.3. These values are the steady state concentrations reached at different times.. It generally took around 2 hrs to reach steady state. Fig 4 shows the effect of current density on the removal efficiency for feed concentrations of 1 ppm, 10 ppm and 100 ppm respectively. All runs conducted at a flow rate of 20 ml/min and pH of 9.8. The removal efficiency (the ratio between the amounts of cyanide oxidized in the cell to the total amount of cyanide fed to the electrolytic cell) increased with an increase in current density. This increase is due to the increased electrode reaction rates and agrees with the results of Ho et al [21] who used stainless steel fibers as electrode materials.

Current efficiency of cyanide removed is defined as the amount of cyanide actually removed divided by the theoretical amount, which should have been removed according to Faraday's law. Fig 5 shows the effect of cell current density on the current efficiency for 1 ppm, 10 ppm and 100 ppm inlet concentrations. The current efficiency decreases with current density due to increase in oxygen evolution.

#### 3.1.2. Effect of flow rate

The effect of flow rate on the outlet concentration of cyanide is shown in Fig 6. All runs were conducted at pH value of 9.8. Also Fig (7) shows that the removal efficiency increases with flow rate because the mass transfer coefficient and its rate increases. It was found that the removal efficiency reaches a maximum at 20 ml/min. and then decreases due to the decrease in residence time inside the anode compartment. Fig 8 shows that the current efficiency increases with flow rate at a constant current density. This is due to the increase of mass transfer rates increase with flow rate increase.

#### 3.1.3. Effect of pH

The effect of pH on the outlet concentration of cyanide is shown in Figs

9&10 which show that the removal efficiency increases with pH for inlet concentrations of 1,10 and 100 ppm. This agrees with Tamura et al [23] who found that the anodic oxidation of cyanide is proportional to the alkalinity of the electrolyte. In strong alkaline solutions, the formation of hydrogen cyanide is prevented and minimal anode gassing is observed, Sawyer and Day [27] found that only cyanide ion is electroactive and that the undissociated acid is inert. They concluded that the rate of anodic oxidation is faster at higher pH. In the lower pH case, a one-electron oxidation mechanism is accepted and cyanogen appears.

At higher pH levels, the limiting currents increase indicating the occurrence of cyanate formation where cyanogen will react rapidly with hydroxide in solution so the removal efficiency increases. When the hydroxide concentration in the bulk solution becomes very low, the limiting current density indicates cyanogen formation. Fig 11 Shows that the current efficiency increases with increase in pH because the anolyte and catholyte are mixed, the pH will rise and any cyanogen appearing in the anolyte will be converted to cyanate so current efficiency increases provided that no other side reactions occur at the anode. These experiments were conducted at a flow rate of 20 ml/min.

### 3.1.4. Effect of cell arrangement:

Fig 12 shows that the vertical arrangement is better than the horizontal arrangement. It is observed that; in the horizontal arrangement the gases accumulate in the cell and it is difficult for gases to go out easily.

### 3.2. Effect of current density on the outlet concentration, removal efficiency and current efficiency of Zinc ion alone

The effect of current density on outlet concentration of zinc is shown in Fig 13. These are the steady state concentration reached at different times. The approach to

steady state takes around 2 hrs. Fig 14 shows that the removal efficiency increases with current density. This increase is due to the increased electrode reaction rates. Fig 15 also shows that the current efficiency decreases with increasing current density. This is due to the increase of hydrogen evolution as current increases.

### 3.3. Effect of operating conditions on the outlet concentration, removal efficiency and current efficiency of both zinc and cyanide ions:

#### 3.3.1. Effect of current density:

Fig 16 shows that the effect of current density on the removal efficiency of cyanide and zinc ions. Removal efficiencies of both cyanide and zinc ions increase with increase in current density. This increase is due to the increase electrode reaction rate. There was no significant change took place for affecting of current density on removal efficiency between each ion alone and both ions with together. This agrees with Ho et al [21] who used stainless steel fibers as electrode materials. Fig 18 shows that the current efficiency of cyanide removal decreases with current density because oxygen evolution increases with current but current efficiency of zinc removal increases with increase in current density because hydrogen evolution is not significant at these conditions.

#### 3.3.2. Effect of flow rate

Fig 17 shows the effect of flow rate on the removal efficiency of zinc and cyanide where it increases with flow rate because the mass transfer rates for zinc and cyanide ions increase with flow rate. This agrees with Lanza and Bertazzoli [35] and agrees with Tissot and Fragniere [25]. There no change took place between the effect of flow rate on removal efficiency for each ion and the effect of flow rate on removal efficiency for both ions. Fig 18 shows that the current efficiency of zinc and cyanide removal

increases with an increase in flow. There was no change between the effect of flow rate on current efficiency for each ion and the effect of flow rate on current efficiency for both ions.

### 3.3.3. Effect of pH

Fig 19 shows that the removal efficiency of cyanide ion increases with an increase in pH while no change took place in removal efficiency of zinc ion with pH increase. For cyanide ; at low pH, a one electron oxidation mechanism is suggested and cyanogen appears. At high pH levels the limiting current increased indicating the formation of cyanate. No change took place between cyanide ion alone or combination with zinc for the effect of pH on removal efficiency. This agrees with Tamura et al [23]. For zinc: at lower pH, the limiting current is higher and removal efficiency increases. As pH increases, a decrease is observed in limiting current and removal efficiency decreases.

### 3.3.4. Effect of Inlet concentration:

Figs 20&21 show that both removal efficiency and current efficiency of zinc and cyanide increase with concentration increases. This agrees with the findings of Ho et al [21] and Christophers[33].

## 4. CONCLUSION

An electrochemical method was employed in the present study to treat the electroplating wastewater. The effect of pH, current density, flow rate and inlet concentrations were investigated. The removal efficiency of zinc and cyanide increased with an increase in current density, flow rates and inlet concentration for both ions. The electrochemical method can be used successfully for oxidation and removal of cyanide ions and also the reduction and separation of zinc ions. It is possible to remove cyanide and zinc ions simultaneously at reasonable current and

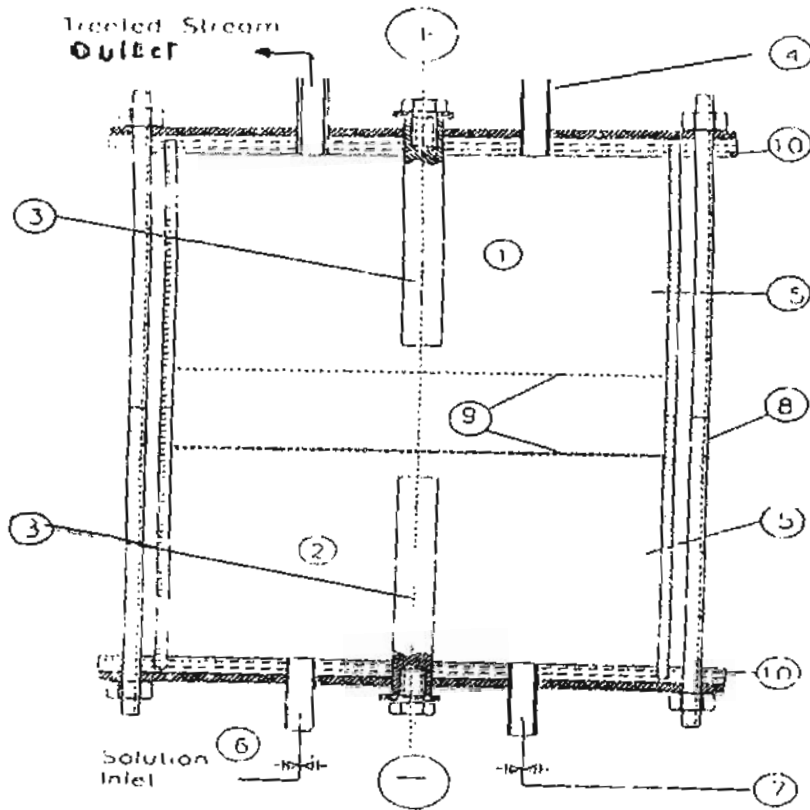
removal efficiencies using the same electrochemical cell.

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|------------------|--------------------------------|--------------------------|-------------|
| 1- Anode         | 2- Cathode                     | 3- Stainless steel rods  | 4- Gas vent |
| 5- Graphic flaks | 6- Needle valve                | 7- Cathode outlet stream |             |
| 8- Tie rod       | 9- Plexi-glass perforated disk | 10-Plexi-glass cover     |             |

Fig 1 Experimental cell

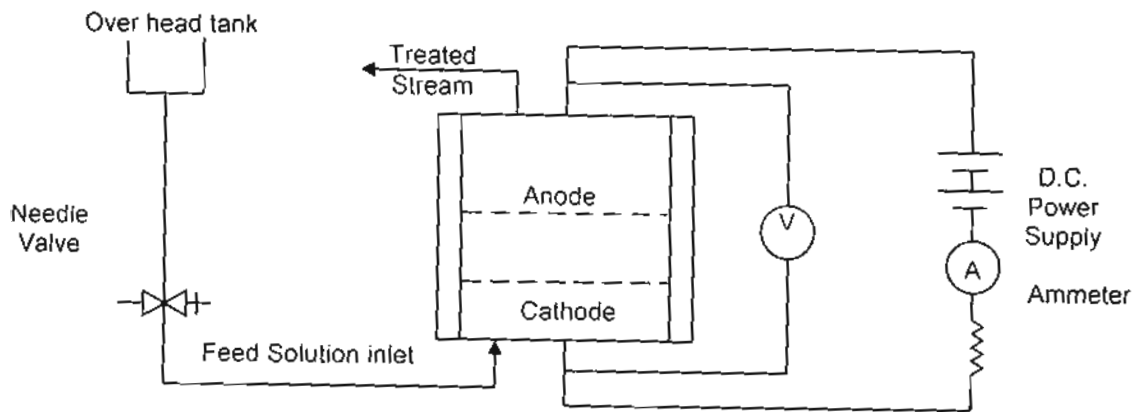
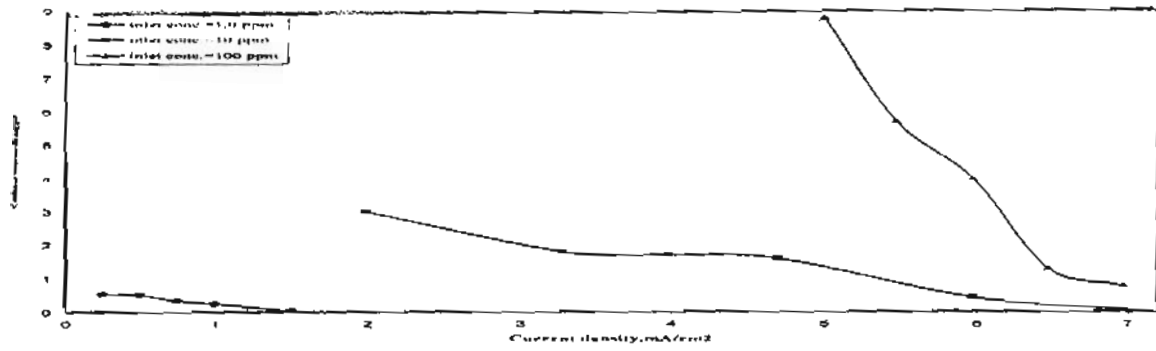
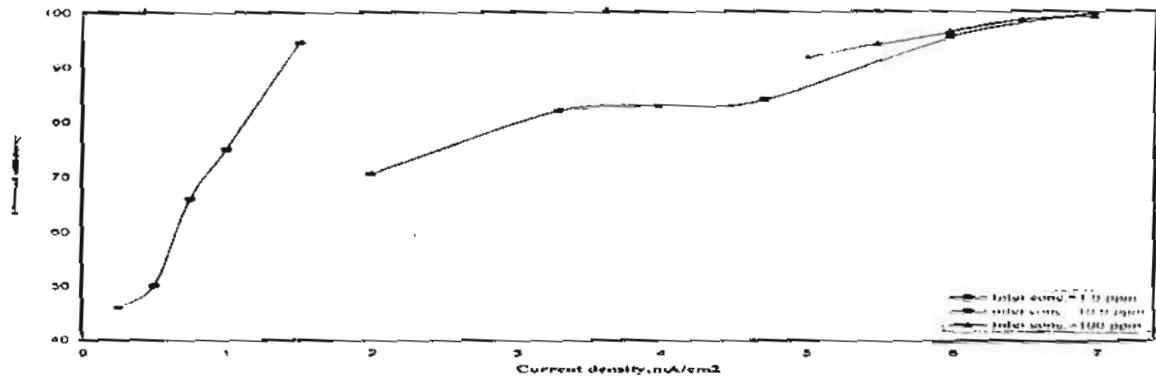


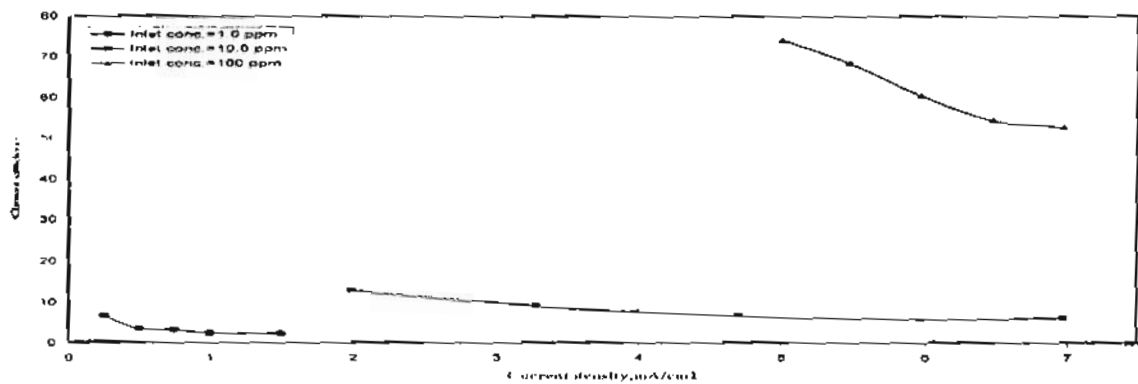
Fig 2 Schematic diagram of experimental apparatus



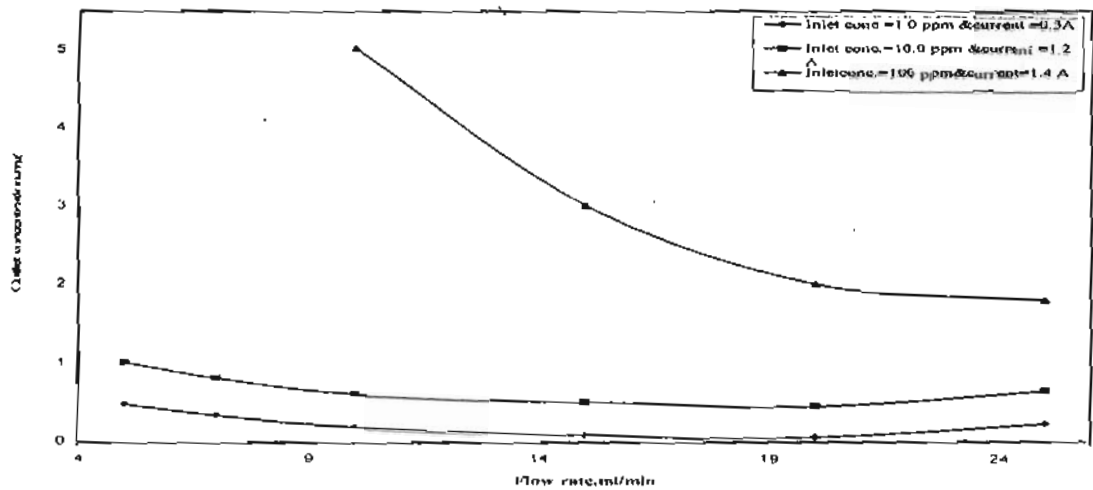
Fig(3) Effect of current density on outlet concentration of cyanide



Fig(4) Effect of current density on removal efficiency of cyanide



Fig(5) Effect of current density on the current efficiency of removal cyanide



Fig(6) Effect of flow rate on the outlet concentration of cyanide

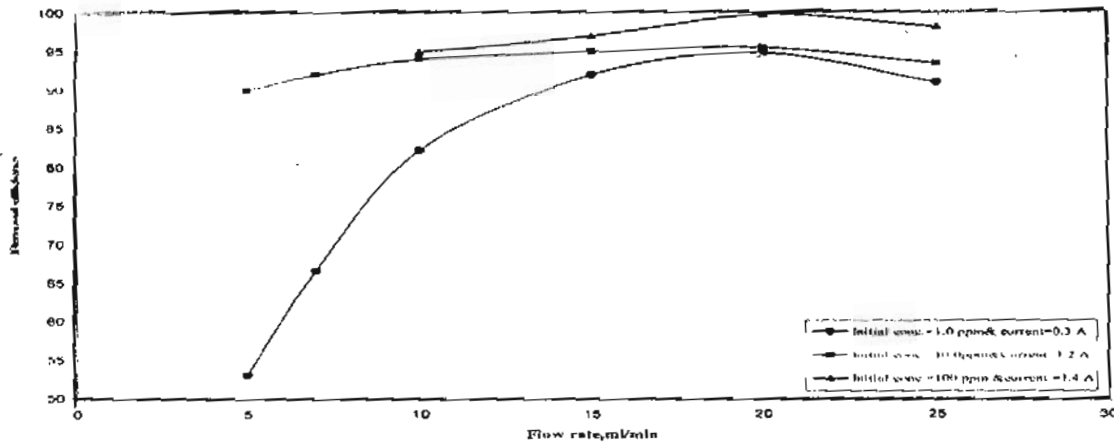
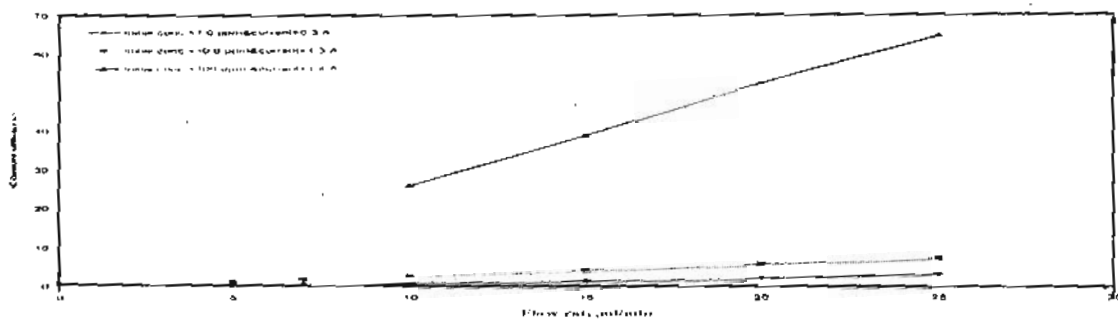
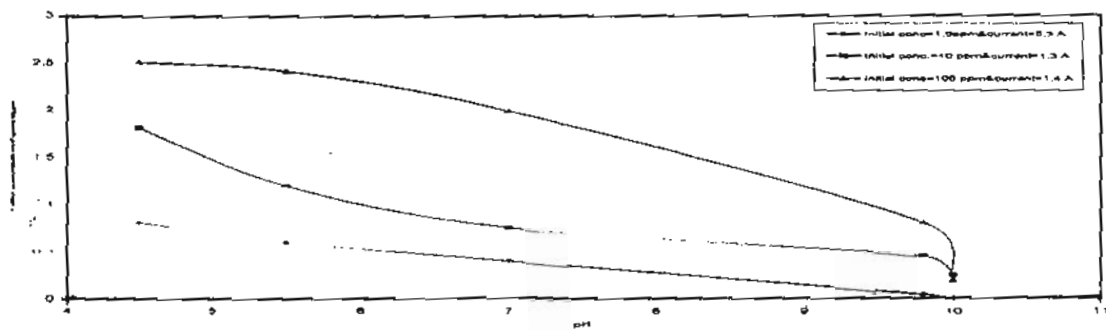


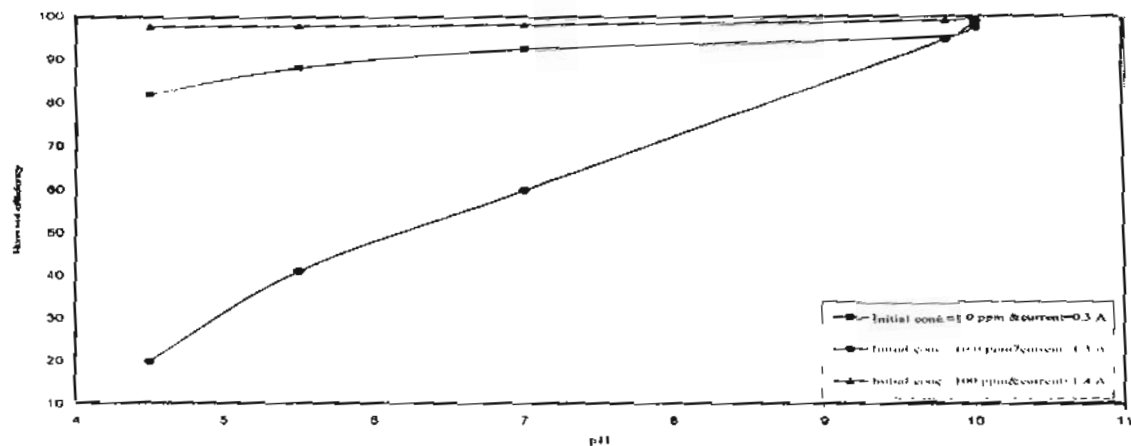
Fig (7) Effect of flow rate on the removal efficiency



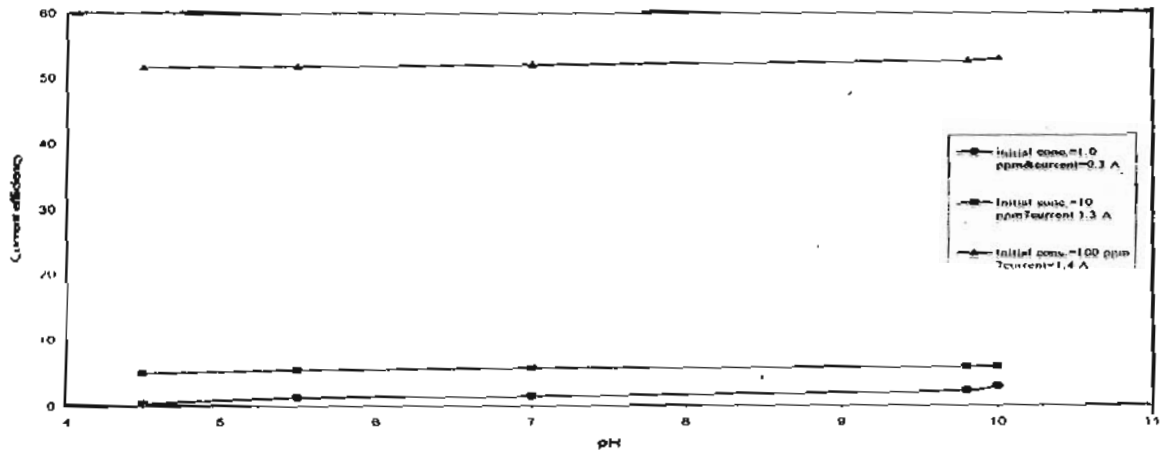
Fig(8) Effect of flow rate on the current efficiency of cyanide removed cyanide



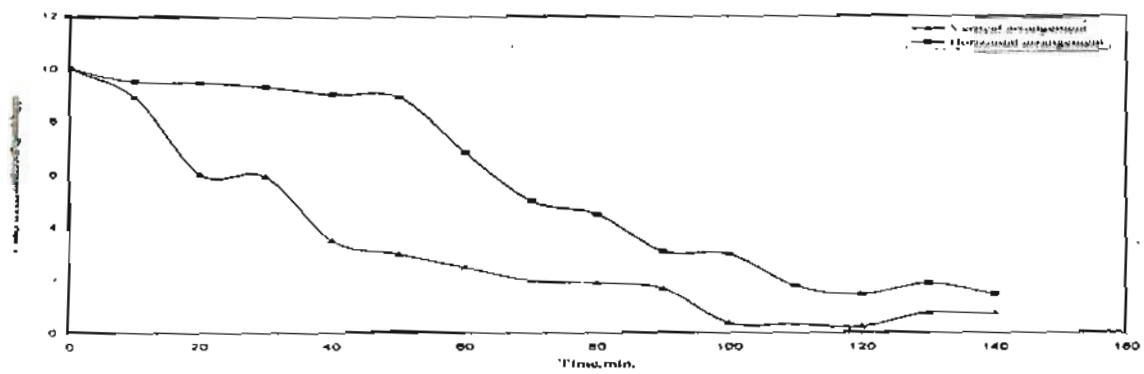
Fig(9) Effect of pH on outer concentration of cyanide



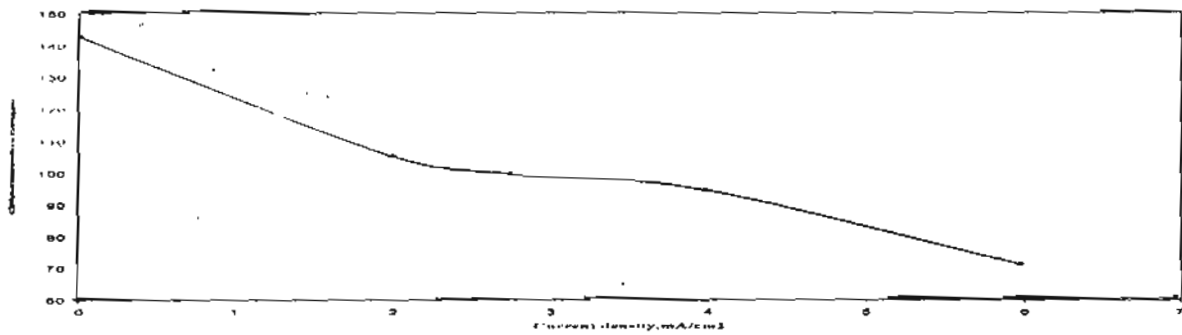
Fig(10) Effect of pH on removal efficiency



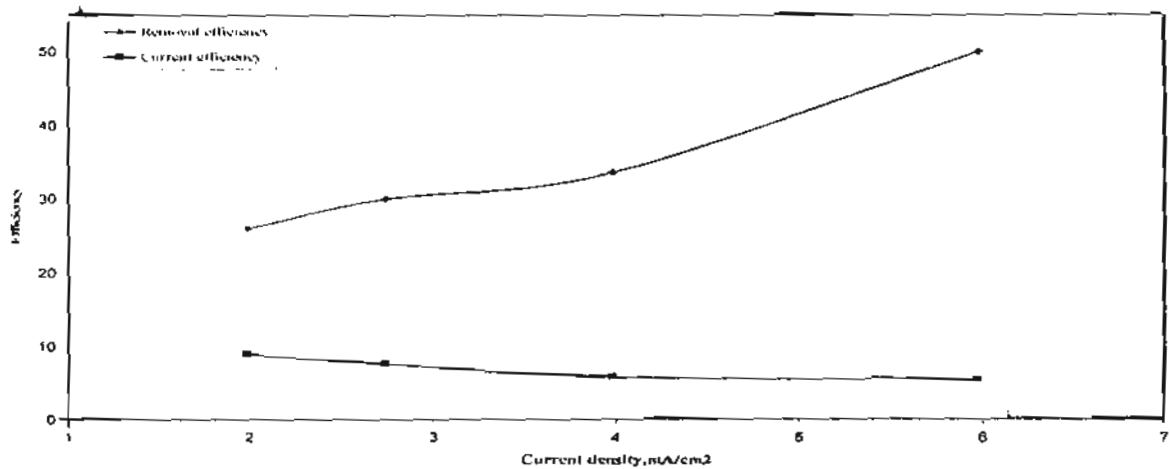
Fig(11) Effect of pH on current efficiency of removed cyanide



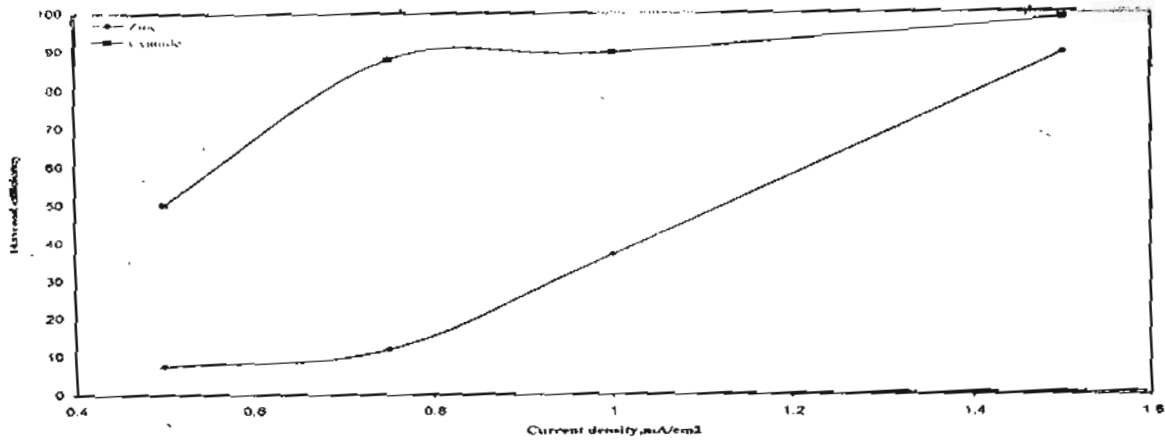
Fig(12) Effect of cell arrangement on the outlet concentration of cyanide



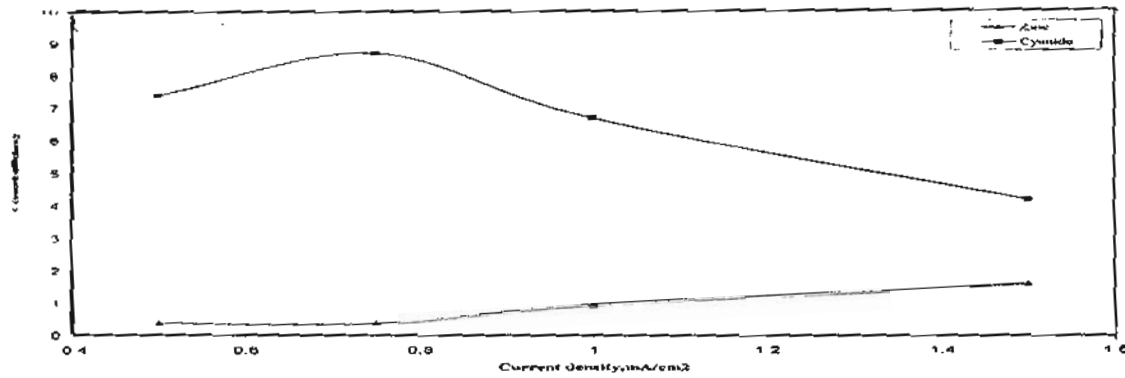
Fig(13) Effect of current density on the outlet concentration of zinc



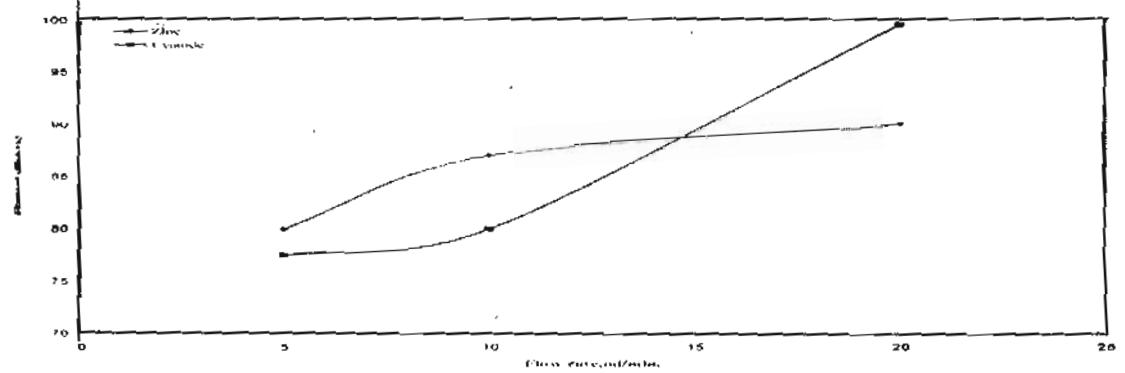
Fig(14) Effect of current density on both removal efficiency of zinc and current efficiency of zinc removed



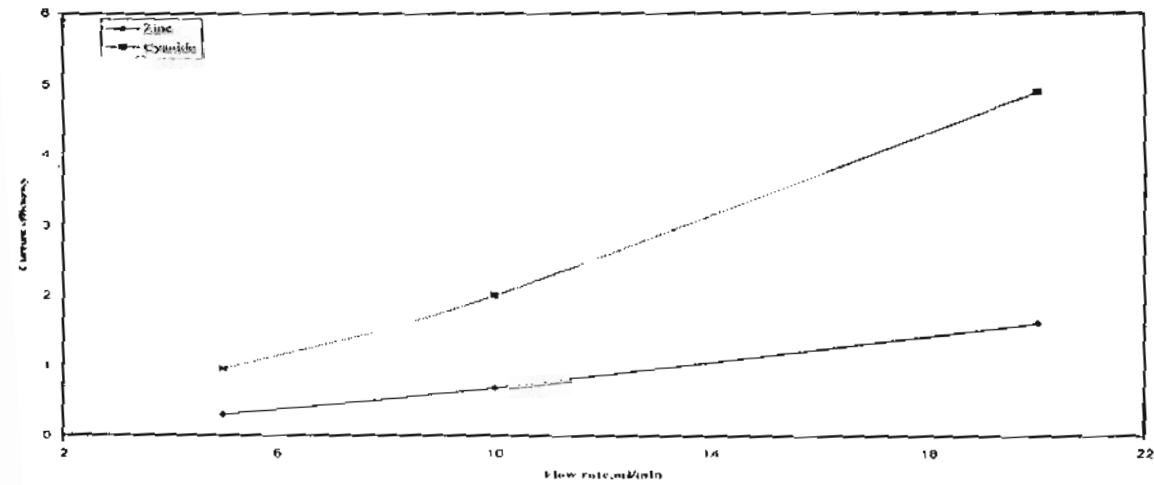
Fig(15) Effect of current density on the removal efficiency of both cyanide and zinc



Fig(16) Effect of current density on the current efficiency of the removal of both zinc and cyanide



Fig(17) Effect of flow rate on the removal efficiency of both zinc and cyanide



Fig(18) Effect of flow rate on the current efficiency of the removal of both cyanide and zinc

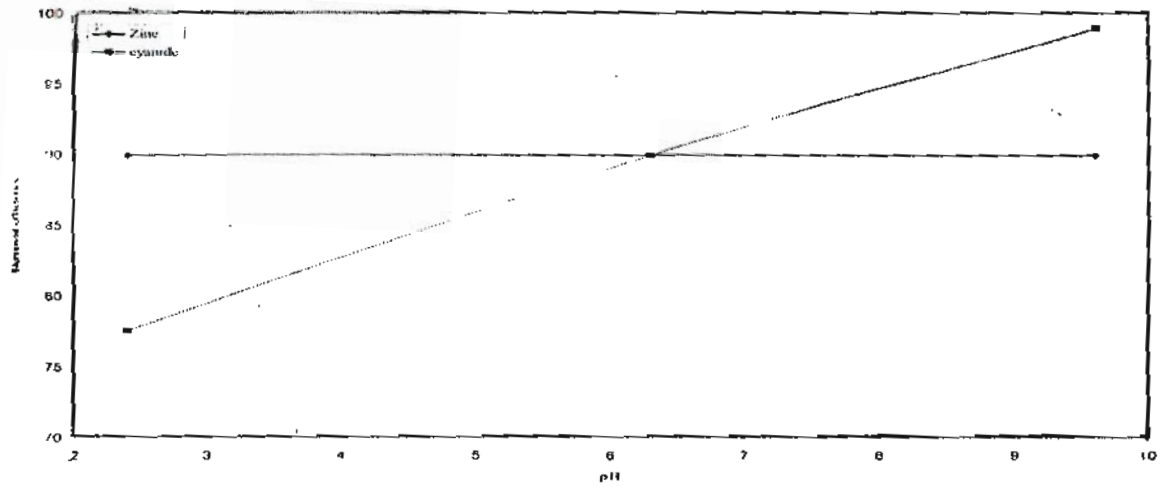


Fig (19) Effect of pH on the removal efficiency of both zinc and cyanide

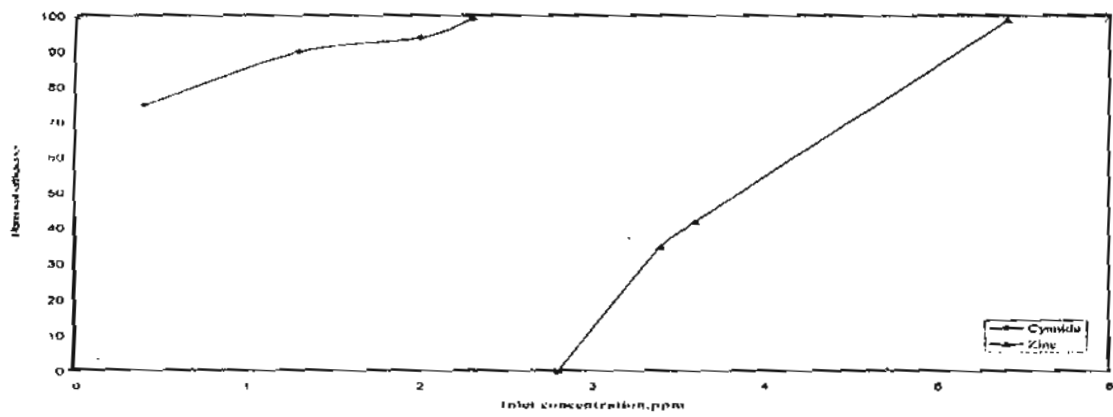


Fig (20) Effect of Inlet concentration on the removal efficiency for zinc and cyanide

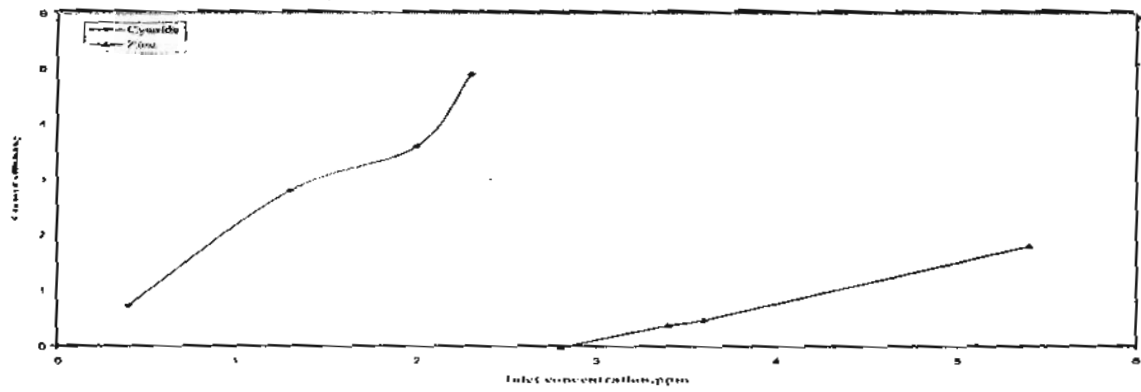


Fig (21) Effect of Inlet concentration on the current efficiency of cyanide and zinc